

joint research centre
EUROPEAN COMMISSION

Annual Report

**Institute for Reference
Materials and Measurements**

1999

Institute for Reference Materials and Measurements Annual Report 1999



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

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This report was produced by Dr. C. Nicholl (editor and layout) and Dr. D. Florian

Inquiries should be addressed to the Commercialisation of Reference Materials and Methods and Scientific Liaison (CSL) Office

Institute for Reference Materials and Measurements, Retieseweg, B-2440 Geel,
Phone +32 14 571 271, Fax +32 14 584 273, E-mail: marketing@irmm.jrc.be

Information on the institute may also be found on the Web page
<http://www.irmm.jrc.be>

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INSTITUTE FOR REFERENCE MATERIALS AND MEASUREMENTS



ANNUAL REPORT 1999



EUROPEAN COMMISSION

JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

Geel, Belgium

Mission

The mission of IRMM is to promote a common European measurement system in support of EU policies, especially internal market, environment, health and consumer protection standards.



Excellent scientific results in support to food safety, international trade, environmental protection and nuclear safety have been produced in the past year. The changes that have been introduced in the latter half of the 1990s including the refocussing of our scientific research lines, the revamping of administrative procedures, the creation of a new unit for marketing and the introduction of total quality management including project management, are now beginning to harvest rewards in terms of scientific excellence.

EUROPEAN SCIENTIFIC AND TECHNOLOGICAL EVOLUTION

Today, Europe produces one third of the world's scientific knowledge and is at the forefront in the chemical and medical research sectors. However, the Union's overall research effort in relation to its gross domestic product has been steadily declining for 10 years and is currently at 1.8% as compared to 2.8% in the United States and 2.9% in Japan. In terms of employment figures, researchers account for only 2.5 in every thousand of the industrial workforce in Europe as compared to 6.7 in the United States and 60 in Japan. Commissioner Philippe Busquin and his cabinet have quickly recognised the need for change and, as a result, a strategy paper entitled "Towards a European Research Area" has been submitted to the Council, the Parliament, the Economic and Social Committee and the Committee of the Regions.

There are nevertheless changes on the horizon for research in Europe and the Commission predicts that the 21st century will be the century for science and technology. The new strategy of the Commission clearly defines what it terms "a common system of scientific and technical reference for policy implementation" wherein IRMM and the JRC as a whole, will play a more than significant role given its institutional proximity to the development of the European Union's policies and its independence of national and private interests. Reference materials and reference measurements will form indispensable cornerstones for the development of this common European measurement system and IRMM, with its specialised facilities, top level personnel and 4 decades experience, will provide sound scientific support and remain available to respond to the requests of the European Union.

THE FUTURE

In accordance with the Amsterdam Treaty IRMM's future will spearhead customer oriented research in the areas of consumer protection, health, environmental protection, reliability of international trade, enlargement of the EU, the safety of nuclear energy and the control of nuclear materials. Its challenges in scientific research will parallel the challenges for the European Union in terms of globalisation with major focus on the removal of barriers to trade, increased competition, reduction of the number of protected sectors and further expansion beyond the present frontiers.

ACKNOWLEDGEMENTS

I would like to express my appreciation and gratitude to research partners, clients, customers and users of our research from industry, public and private research institutions in the Member States, the EFTA (European Free Trade Association) countries, pre-accession countries, Switzerland, USA, Canada, Japan, Australia, China, Chile and international organisations for fruitful collaborations, then to colleagues of the Direction General JRC and other Commission Services for continuous support during the year.

Finally I wish to sincerely thank the staff of IRMM for performing excellent research, fine administration and for showing flexibility and dedication towards the changes at IRMM and within the JRC as a whole.



A handwritten signature in dark ink, appearing to read 'M. Grasserbauer', written in a cursive style.

Manfred Grasserbauer

Director

Institute for Reference Materials and Measurements

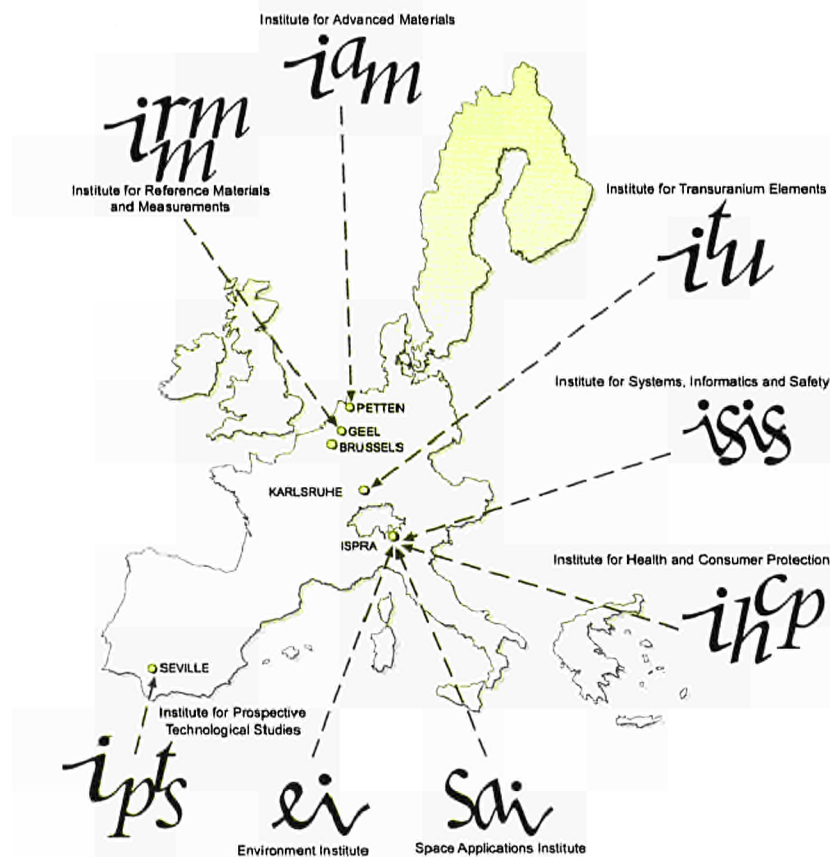
EUROPEAN COMMISSION - JOINT RESEARCH CENTRE

BACKGROUND

The JRC is the European Commission's scientific and technical research laboratory. Over the years it has developed special skills and unique tools to provide independent and Europe-wide expertise to better understand the links between technology, the economy and society.

MISSION

"The mission of the JRC is to provide consumer-driven scientific and technical support for the conception, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a centre of science and technology (S/T) reference for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, private or national".



SIZE AND FUNDING

The JRC employs about 2380 staff under different schemes and uses a budget of over 300 Million EURO per year stemming from the European Research budget and from competitive income.

Five separate sites, located in Belgium, Germany, Italy, the Netherlands and Spain, house eight different institutes, each with its own focus of expertise. The JRC headquarters is located in Brussels, with the Programmes Directorate serving as a link between the institutes and the policy makers. The Directorate coordinates the research performed by the eight institutes and helps to ensure its quality by interacting with the international scientific community and industry.

INSTITUTE FOR REFERENCE MATERIALS AND MEASUREMENTS

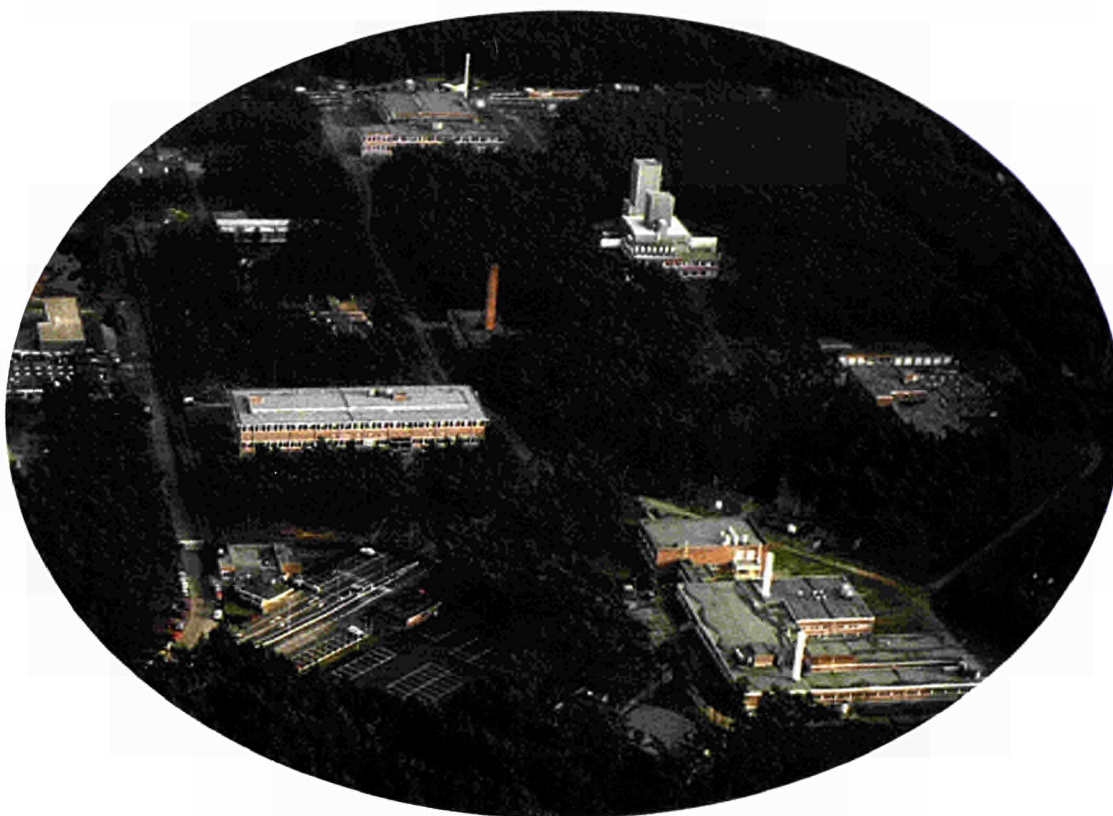
BACKGROUND

The IRMM is one of the eight institutes of the Joint Research Centre and is situated in Belgium some 80 km north-east of Brussels. Founded in 1957 and located in the tranquil surroundings of the Flanders countryside, the IRMM consists of 10 buildings located within a 42 hectare site.

With 40 years experience in specialised research, the careful construction and development of unique facilities together with a focused training and nurturing of excellent staff, the Institute for Reference Materials and Measurements has, in many ways, evolved into becoming the Trans-national Measurement Institute (TMI) of the European Commission - analogous to the National Measurement Institutes (NMI) of the Member States. In this function it directly supports the Member States by providing reference materials and measurements.

MISSION

"The mission of IRMM is to promote a common European measurement system in support of EU policies, especially internal market, environment, health and consumer protection standards".



SIZE AND FUNDING

IRMM has a total staff count of 214 persons, executes an annual budget of 30 million Euro of which 8.5% is earned through competitive means.

IRMM serves the policies of the European Union by delivering scientific/technical products and services for the European Commission's Directorate Generals (DGs), international organisations, Member States and their institutions, and industry including SMEs.

THE IRMM MANAGEMENT STAFF*



Prof. Dr. Manfred Grasserbauer
Director
Tel.: +32(0)14 571-292
Fax: +32(0)14 584-294
manfred.grasserbauer@jrc.org



Dr. Hermann Weigmann
Neutron Physics
Tel.: +32(0)14 571 475
Fax: +32(0)14 591 980
hermann.weigmann@irmm.jrc.be



Michael-Francis Fahy, M.Sc. MBA
Management Support
Tel.: +32(0)14 571 316
Fax: +32(0)14 584 273
michael-francis.fahy@jrc.org



Dietmar F.G. Reher, M.Sc.
Radionuclide Metrology
Tel.: +32(0)14 571 263
Fax: +32(0)14 584 273
dietmar.reher@irmm.jrc.be



Dr. Doris Florian
Marketing and Scientific Liaison
Tel.: +32(0)14 571 272
Fax: +32(0)14 584 273
doris.florian@irmm.jrc.be



Dr. Richard Ross
Informatics and Electronics
Tel.: +32(0)14 571 478
Fax: +32(0)14 591 980
richard.ross@irmm.jrc.be



Dr. Jean Pauwels
Reference Materials
Tel.: +32(0)14 571 722
Fax: +32(0)14 590 406
jean.pauwels@irmm.jrc.be



Luc Peeters (Civil Engineer)
Radiation Protection and Security
Tel.: +32(0)14 571 282
Fax: +32(0)14 584 273
luc.peeters@irmm.jrc.be



Prof. Dr. Adela Rodriguez
Chemical Reference Measurements
Tel.: +32(0)14 571 200
Fax: +32(0)14 584 273
adela.rodriguez@irmm.jrc.be



Dr. Jean-Claude Spirlet
Quality Management
Tel.: +32(0)14 571 767
Fax: +32(0)14 584 273
jean-claude.spirlet@irmm.jrc.be



Dr. Philip Taylor
Isotopic Measurements
Tel.: +32(0)14 571 605
Fax: +32(0)14 591 978
philip.taylor@irmm.jrc.be



Prof. Dr. Paul De Bièvre
Advisor for Metrology in Chemistry
Tel.: +32(0)14 571 608
Fax: +32(0)14 591 978
paul.de.bievre@skynet.be



Dr. Roger Wellum
Nuclear Safeguards
Tel.: +32(0)14 571 607
Fax: +32(0)14 591 978
roger.wellum@irmm.jrc.be



Prof. Dr. Achiel Deruytter
Advisor for Neutron Physics
Tel.: +32(0)14 571 314
Fax: +32(0)14 584 273

* Status on 31st December 1999

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SCIENTIFIC PROFILE IN 1999

1. SCIENTIFIC HIGHLIGHTS

Post-mortem Testing for Bovine Spongiform Encephalopathies (BSE):



IRMM completed the preparation and distribution of over 14000 samples to 4 different companies, the supervision of the measurements and the evaluation of some 5290 results. Three rapid post-mortem BSE tests showed remarkable effectiveness and achieved a 100% success rate in distinguishing animals clinically affected with BSE from healthy animals. This breakthrough in BSE research has already been published in 'Nature'. Projects were also completed for heat treatment procedures for BSE decontamination and for the detection of animal meal in vegetarian feeding.

Proficiency Testing Samples to detect Genetically Modified (GM) Foods:



1000 units (1g) each of 1 % and 5 % modified Roundup-Ready® Soya and Bt-176 Maize respectively have been prepared for the validation of a quantitative PCR (polymerase chain reaction) method to detect GM foods.

New Serum Panel for Cortisol:

In collaboration with the Federation of Clinical Chemistry (IFCC) the production of a new serum reference panel (CRM IRMM/IFCC 451) to standardise cortisol test systems has been completed in 1999. Cortisol has been quantified in a panel of 34 native serum samples obtained from 18 female and 16 male blood donors with a concentrations range from 82.7 - 764.2 nmol/L.

Response to Dioxin crisis:



With the advent of the 1999 dioxin crisis, sales of reference materials (BCR) for PCB (Polychlorinated Biphenyl) analysis rose, in some cases, by several hundred percent. To provide further support an immediate action was undertaken by IRMM to prepare and certify (doped) pork Reference Materials (RMs) and to make this set of Certified Reference Materials (CRMs) in pork fat matrix available for Quality Control purposes in laboratories EU-wide.

Management of BCR Certified Reference Materials:

In 1999, a total of 8181 samples have been sold and distributed through 1998 processed orders. Stability monitorings were initiated, both internally (NAA, GCMS, etc) and externally, for the control of more than 100 existing BCR and/or assimilated IRMM reference materials. In accordance to EN 10045-2, IRMM certified 16 new CRM batches for the impact toughness of steel testing (charpy specimens) and participated successfully in an international evaluation of charpy reference specimens involving Japanese, French and American metrology institutes.

Antibiotics in Domestic Meat Supplies:



The problem of antibiotics in domestic meats has prompted intervention by the EC. This new project, in support to EC Directive 70/524/CEE and EC Regulation 2821/98, commenced with literature research and collaboration with the University of Liège (Department of Veterinary Medicine). Two families of antibiotics (macrolides and quinolones) were selected and commercially available standards were acquired. The separation of 5 quinolone antibiotics by CZE-DAD was carried out successfully and the same compounds were submitted for ESI-MS/MS fragmentation studies. Excellent results with a low limit of detection were achieved.

Radionuclides in Food:



The enlargement of the EU has prompted an increasing number of requests from national and international bodies (e.g. DG External Relations and DG Research) for food reference materials that are certified for radionuclides (Council Directive 96/29/EURATOM). At IRMM a reference method to determine trace nuclear impurities in food materials is being developed using ICP-MS. In 1999, matrix effects, column capacity and elution profiles were examined, a new separation method based upon extraction chromatography was developed, alpha spectrometry analyses

were performed and a detailed study of the sorption selectivities of Th, U, Pu and Am from water was completed.

Support to International Trading Systems:

IRMM's International Measurement Evaluation Programme (IMEP) which shows the state of international equivalence of chemical measurements hit the headlines in 1999. The first ever BIPM (Bureau International des Poids et Mesures) Key Comparison in the area of chemical measurements was successfully completed with IRMM as the co-ordinating laboratory. IMEP rounds -9 (trace elements in water) and -11 (metals in car exhaust catalysts) were completed (with over 235 participant laboratories from over 40 different countries). New IMEP rounds were launched, namely IMEP-13 (metals in polymer) and IMEP-14 (metals in sediment) and the latter has been requested by the Consultative Committee on Amount of Substance (CCQM) of the BIPM to be used in an inter-comparison study.

Safeguarding the Environment:

In collaboration with the official USA Safeguards Laboratory in New Brunswick, USA, IRMM became engaged in the production of a set of global isotopic reference materials for the nuclear accounting of uranium.

Production of the world's first Primary Isotopic Gas Standards (PIGS) for measuring SI (International System of Units) traceable isotope ratios in food and in clinical, atmospheric and environmental chemistry continued. Progress was achieved with the completion of the SF₆ certificate, the preparation of a carbon PIGS (CF₄) which was bottled at Messer Griesheim GmbH (Germany) and measurements of CF₄ PIGS on the "Avogadro II amount comparator" were completed.

Neutron Measurements for Reactor and Radiation Safety:

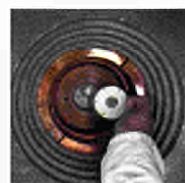
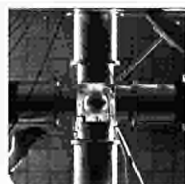
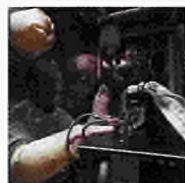
To evaluate reactor and radiation safety, activation cross section measurements for short-lived products are very important. In 1999, cross sections for 30 short-lived products were measured between 16 and 21 MeV at IRMM and for ⁹⁹Tc, measurements between 9 and 12 MeV were conducted at the Forschungszentrum Jülich (Germany). This is the second measurement campaign in collaboration with Argonne National Laboratory (USA) on isotopes of Cr, Cu, Mo, Se, Sr, Zn, Zr, V, and Tc.

Neutron Measurements for Waste Transmutation:

Representing a major environmental concern, long-lived fission products and actinides present in nuclear waste are under investigation for a possible transmutation into short-lived or stable products by means of neutron irradiation. For ⁹⁹Tc(n,T), (n,γ) cross section measurements, the analysis of transmission data was completed (E-range: thermal to 100 keV) and published and the resonance parameters (up to 10 MeV) will be sent to the NEA (Nuclear Energy Agency (OECD)) Data Bank. Furthermore ²³⁷Np(n,T), (n,γ) cross section measurements have been completed and the data analysis is ongoing.

IRMM to reproduce International Ionisation Chamber:

For radioactivity measurements the most important 'key-comparison' is the Système International de Référence (SIR). This consists of a commercial ionisation chamber that has been calibrated with about 500 world-wide primary standards of radioactivity for about 60 radionuclides. The estimated effort world-wide for this chamber is about 200 man-years or some 10 - 20 million EURO. IRMM, in collaboration with the National Physical Laboratory (UK), launched a project for the Consultative Committee for Ionizing Radiation (CCRI) to rescue the SIR in case of diminution. An ionisation chamber that is reproducible in its characteristics has been conceived, simulations for parameters, such as physical dimensions, materials, type and pressure of gas, were carried out by means of EGS4. Comparison with the efficiency of existing chambers was successful and a first draft design is being prepared and the patent has already been filed.



2. SCIENTIFIC CO-OPERATION AGREEMENTS

CO-OPERATIONS WITH THE DIRECTORATES GENERAL

IRMM's direct support to EU policies can be represented through an official collaboration network with various Directorates General of the European Commission, namely:

Agriculture, Energy, Enterprise, Environment, External Relations, Research, Health and Consumer Protection, Information Society, Internal Market, Taxation and Customs Union.

CO-OPERATIONS WITHIN THE EU

Air Liquide, Paris, France

BASF, Ludwigshafen, Germany

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany

Bureau International des Poids et Mesures (BIPM), Paris, France

Compagnie Générale des Matières Nucléaires (COGEMA), France

Carso, Lyon, France

Comité Européen de Normalisation (CEN), France

Central Science Laboratory (CSL), United Kingdom

Centro de Investigaciones Energéticas Medioambientales y Tecnológicas (CIEMAT), Madrid, Spain

Centro Nazionale per i Materiali di Riferimento (CNMR), Rome, Italy

Cogne Acciai Speciali, Aosta, Italy

Commissariat à l'Energie Atomique (CEA), France

Comité de Liaison Européen de l'Industrie de la Parfumerie, des Produits Cosmétiques et de Toilette (COLIPA), Brussels, Belgium

Dr. Ehrenstorfer GmbH, Wesel, Germany

EQAL, København, Denmark

EURACHEM, Helsinki, Finland

EUROMET, Paris, France

European Accreditation of Laboratories (EA), Berlin, Germany

Eurofins, Nantes, France

Finigan MAT, Bremen, Germany

Forschungszentrum (FZJ), Jülich, Germany

Forschungszentrum (FZK), Karlsruhe, Germany

Hamburger Synchrotronstrahlungslabor, Hamburg, Germany

Hellenic Institute for Metrology (HIM), Thessaloniki, Greece

Hitec, Karlsruhe, Germany

Institute for Agrobiotechnology (IFA), Tulln, Austria

Institut Laue-Langevin (ILL), Grenoble, France

International Atomic Energy Agency (IAEA), Vienna, Austria

Istituto di Metrologia Gustavo Colonnetti (IMGC), Torino, Italy

Istituto Superiore di Sanità (ISS), Rome, Italy

Laboratoire National d'Essais (LNE), Trappes, France

Laboratoire Primaire des Rayonnements Ionisants (LPRI), Saclay, France

Laboratory of the Government Chemist (LGC), Teddington, United Kingdom

Merck, Darmstadt, Germany

Messer Griesheim GmbH, Duisburg, Germany

Montanuniversität Leoben, Austria

National Physical Laboratory (NPL), London, United Kingdom

Nederlands Meetinstituut (NMI) Delft, The Netherlands

Organisation for Economic Cooperation and Development (OECD), France

Physikalisch Technische Bundesanstalt (PTB), Braunschweig, Germany

Promochem, Oberwesel, Germany

Roche-Böhringer, Mannheim, Germany

Slovak Institute of Metrology, Slovakia

Swedish National Testing and Research Institute (SP) Borås, Gothenburg, Sweden

Studiecentrum voor Kernenergie (SCK/CEN), Mol, Belgium

TNO, Zeist, The Netherlands

University of Antwerp, Belgium

University of Barcelona, Spain

University of Bonn, Germany

University of Jyväskylä, Finland

University of Köln, Germany

University of Leuven, Belgium

University of Oxford, United Kingdom

VDA, Frankfurt, Germany

Vrije Universiteit Amsterdam, The Netherlands

Vlaamse Instelling voor Technologisch Onderzoek (VITO), Mol, Belgium

Wagner Analysen Technik und Vertrieb, Bremen, Germany

CO-OPERATIONS OUTSIDE THE EU

In order to support enlargement and globalisation, a stronghold of collaborations with international companies, institutions and organisations is of paramount importance. IRMM's partners outside the EU include:

Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials (ABACC), San Paulo, Brazil

Asahi, Tokyo, Japan

CONICYT (Chilean Commision for Science and Technology), Chile

EMPA, St. Gallen, Switzerland

Fluka Chemie AG, Switzerland

International Nuclear Target Development Society, Deer Harbour, USA

International Committee for Radionuclide Metrology, Washington, USA

International Federation of Clinical Chemistry (IFCC), Ontario, Canada

International Standards Organisation (ISO), Geneva, Switzerland

Japan Atomic Energy Research Institute (JAERI), Japan

Japanese Committee for Clinical Laboratory Standards, Japan

Korean Atomic Energy Research Institute (KAERI), Korea

Los Alamos National Laboratory, USA

MFA Research Institute for Technical Physics and Materials Science, Budapest, Hungary

Monsanto, USA

National Institute for Standards and Technology (NIST), Washington, USA

National Measurement Laboratory, Sydney, Australia

National Research Laboratory for Metrology, Tsukuba, Japan

New Brunswick Laboratory, Chicago, USA

Novartis Seeds, Basel, Switzerland

Nuclear Metrology Laboratory, Moscow, Russia

Oakridge National Laboratory, Tennessee, USA

Power Reactor and Nuclear Fuel Development Co-operation, Japan

Research Centre for Certified Reference Materials, Beijing, China

Qinghai Institute of Salt Lakes, China

Swiss Federal Office of Metrology, Wabern, Switzerland

3. INTERNATIONAL SCIENTIFIC RECOGNITION

The promotion of a common European measurement system in support of EU policies depends not only on achieving excellent scientific results, forming a stronghold of collaboration agreements but also on the procurement of international scientific recognition.

ACTIVE INVOLVEMENT IN INTERNATIONAL ORGANISATIONS

BIPM (Bureau International des Poids et Mesures)/VIPM/CCRI (II)
CCRI (Comité Consultatif des Rayonnements Ionisants)
CCQM (Comité Consultatif pour la Quantité de Matière)
CITAC (Co-Operation on International Traceability in Analytical Chemistry)
EURACHEM (Analytical Chemistry in Europe)
EURACHEM Uncertainty Group
EURACHEM Working Group "Proficiency testing"
EUROMET (European Organisation of Metrology)
ISO-REMCO Committee
ISO-REMCO Task Group 1 "Hierarchy"
ISO-REMCO Task Group 4 "Accreditation"
ISO-REMCO Task Group 5 "Sampling"
ISO-REMCO Task Group 6 "Transportation and Distribution of RMs"
IUPAC (International Union of Pure and Applied Chemistry)
EEE-PT (Eurachem - Eurolab - Euromet Proficiency Testing)
ESARDA (European Safeguards Research and Development Association)
Working Group for Low Enriched Uranium Plants
Working Group on Techniques and Standards for Non-Destructive Analysis
IAEA (International Atomic Energy Agency)
Consultant Committee on Reference Materials & Analytical Quality of Isotopic Analysis
ICRM (International Committee for Radionuclide Metrology)
OECD (Organisation for Economic Cooperation and Development) - NEA (Nuclear Energy Agency)
EFF (European Fusion File) Project
JEF (Joint European File) Project
OECD-NEA/NSC

EDITORIAL BOARDS OF SCIENTIFIC JOURNALS

Fresenius Journal of Analytical Chemistry
Japanese Journal of Analytical Sciences
Accreditation and Quality Assurance in Analytical Chemistry
Journal of Analytical Atomic Spectrometry

MEMBERSHIPS IN SCIENTIFIC BOARDS

Chairman of Scientific Council and Member of the Board of Governors of the GSF Research Centre for Environment and Health, Munich

Scientific Council of the Institute for Spectrochemistry and Applied Spectroscopy, Dortmund

Advisory Board of the Reactor Institute Delft

Chairman of "Europe Workshop on Detection Methods for Novel Food Derived from GMO", Brussels

Chairman of "3rd Euroconference on Environmental Analytical Chemistry", Chalkidike 1999

"6th Conference on Hyphenated Techniques in Chromatography" (Member of the Scientific Board)

"International Symposium on Environmental Analytical Chemistry" (Member of the Scientific Board)

"ICRM-99 Conference"

MEMBERSHIPS IN SCIENTIFIC COMMITTEES

SANCO Working Party "Methods of Chemical Analysis for Cosmetics" (Chairperson)

OECD-NEA Nuclear Science Committee

International Nuclear Data Committee

ESARDA Working group on Destructive Analysis (Secretary)

ESARDA Working Group on Non-Destructive Analysis

International Nuclear Target Development Society

IUPAC Subcommittee Isotope Abundance Measurements (Chairperson)

Member of the Eurachem Uncertainty Working Group

IUPAC Analytical Chemistry Division (Past-President)

IUPAC Commission Atomic Weights and Isotope Abundances

IUPAC Committee on Chemistry and Industry

International Committee for Radionuclide Metrology, ICRM (Secretary)

Committee on Chemistry and Industry of IUPAC

EC at EUROMET-Ionising Radiation and Radioactivity

NETWORKS

European Network for the Development of Detection and Identification Methods of GMOs in Food and Food-Ingredients

Validation and Standardisation of Diagnostic PCR for Detection of Food Borne Pathogens

Clinical Reference Materials Network with the International Federation for Clinical Chemistry (IFCC)

Networks of Specific Certified Reference Materials

Joint European Programme for Primary Isotopic Measurements

EUROMET-BIPM Network for Chemical and Radioactivity Measurements

Network for Neutron Data Generation

Network of Metrology and Quality Assurance for Nuclear Safeguards

Speciation 21 Network - Preparing for the 21st century

Thorium Network

Network of Underground Laboratories

MANAGERIAL HIGHLIGHTS IN 1999

Tackling Administrative Procedures:

A new software package (INFIMA) was installed over the year end period. The package will cover purchase, finance, sales, inventory, contracts, and allow for the integrated flow of documents/procedures for all transactions. It brings together the existing stand-alone procedures in an integrated way. This will eliminate much duplication and delay while providing greater openness to users. Additionally a "régie d'avance système" relating to the sales of BCR Reference Materials was successfully negotiated during 1999 and this will greatly simplify/streamline the administrative procedures involved.

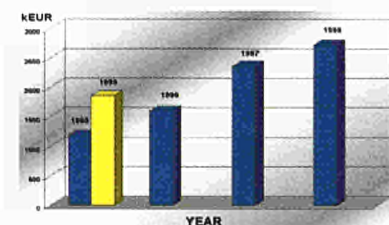
Reforming the Budget Plan:

Budgetary procedures were formalised. Whereas Unit budgets were previously settled on a general, indicative basis, the 1999 process was marked by intensive examination/discussion of the needs of each Unit leading to a precise budget plan agreed and signed by the Director and each Unit Head. This document became the basic budget control tool for all purchases. The process proved very successful and will be repeated in future years.

The allocation of the budget for 1999 was as follows:



In this, the first year of the Fifth Framework Programme (FP5), competitive income reached 1818 kEURO which, although lower than 1998, was an increase compared to the first year of FP4.



Expansion of IRMM Infrastructure:

1999 marked a heavy period of infrastructural investment with four major building extensions getting underway simultaneously. Furthermore, design/planning for a new BCR refrigerated warehouse also continued during the year.

Staff Matters:

The IRMM staff age profile resulted in an unprecedented number of retirements/departures during the year. In all, some 34 statutory staff and 8 fellows left the Institute. The staff status as on 31.12.1999 is depicted in the pie charts below. By comparison with 1998 the total staff number reduced by 16 persons.

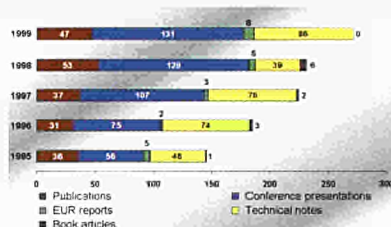


A new staff training procedure was implemented according to Total Quality Management (TQM) and a new individual training form was distributed to staff for improved planning of training activities and career development of staff from 2000 on. Staff participation in training activities increased almost twofold as compared to 1998.

Marketing and Scientific Liaison:

The Unit intensified its activities for competitive activities, international communication and public relations. The Internet homepage (<http://www.irmm.jrc.be>) was updated every two weeks leading to approximately 2400 accesses per month which is a two fold increase compared to just one year ago, the IRMM Annual Report 1998 and a new series of fliers describing the Institute's activities were produced. Support was given to the Units on marketing as well as coordinating project management issues including the introduction of milestones, deliverables, annual resource allocation and regular project review meetings. Two international workshops were organised, 185 visitors hosted and 17 expert lectures arranged. In addition, several events were jointly organised with other JRC Institutes such as the launch of the Fifth Framework Programme in Essen, Germany.

IRMM's modern library facility has co-ordinated the registration of all scientific publications and the 1999 statistics below clearly show an increase in the production and dissemination of scientific results:



Informatics and Electronics:

A new Unit Head was recruited from JET, Culham and appointed on 1st October 1999. During the year the new computer network became fully operational providing 1316 network sockets of which some 600 are already enabled, supporting about 400 devices (mainly PCs).

A central Oracle Server has been installed to support the new database for the sales of Reference Materials, and other Oracle applications. PC Support was extremely active with about 85 new PCs been installed and operating on Windows NT 4.0, and the conversion of the older PCs from Windows 95 has continued throughout 1999.

The electronics and data acquisition sector also made significant progress with the development of the 2000 data acquisition system, using the Modular MultiParameter Multiplexer (MMPM) as the front-end electronics, to which Time Coders and ADC's can be connected. Finally the Unit has been responsible for the co-ordination of a successful millennium rollover.

Total Quality Management:

The implementation of a Total Quality Management (TQM) system based on the model "continuous improvement to business of excellence" and the ISO 9000 standard made significant progress. The documentation (quality manual, procedures) was adapted and the quality manual has now been modified in compliance with "revision 2000" of the ISO 9001 standard. Follow-up on customer complaints led to corrective and preventive actions to improve the quality of reference materials. The first self-assessment exercise according to the EFQM model commenced in 1999. A Reporting Team described the actual situation according to the EFQM criteria. The Assessment Team in charge of the self-assessment and scoring was nominated and received the appropriate assessor training. The exercise will be completed by March 2000. Training in TQM (ISO 9000 and EFQM model) of all employees has started and will be continued during the year 2000. The complete implementation of TQM is targeted for the end of 2000.

**JRC CENTRE
FOR REFERENCE
MATERIALS
AND CHEMICAL
REFERENCE
MEASUREMENTS**



JRC CENTRE FOR REFERENCE MATERIALS

Reference materials constitute an essential tool in achieving comparability and traceability of measurements. When applied to the development of scientific and industrial research, certified reference materials enable the optimisation of industrial processes, the protection of public health and our environment, the promotion of international trade and the implementation and standardisation of European legislation.

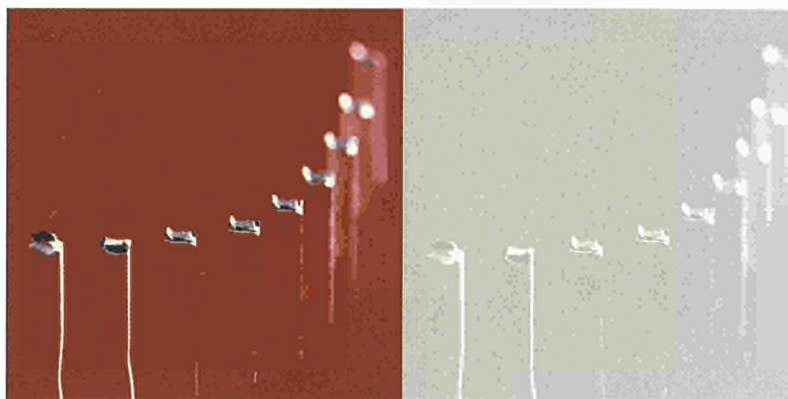
Second only to the National Institute for Standards and Technology (NIST) in USA, the IRMM, in collaboration with the EC's Directorate General Research (Programme Measurements and Testing, Infrastructure), is at the forefront for the production, certification and distribution of Certified Reference Materials (CRMs). Since 1995, the IRMM has shared the responsibility for the production and certification of BCR (Bureau Communautaire de Référence - BCR® is a trademark of IRMM) CRMs with DG Research and since then has taken over full responsibility for the stock management (including storage, stability monitoring, distribution and replacement of exhausted BCR CRMs). At present, IRMM offers over 400 different BCR® CRMs, with a total stock load of some 500,000 samples. The reference materials produced, certified and distributed by IRMM embrace a wide range of fields ranging from agriculture, food and clinical chemistry to industry and environmental protection.

These materials are produced and certified in accordance with internationally accepted rules laid down in guides published by BCR, the International Standards Organisation (ISO) and the World Health Organisation (WHO).



IRMM Certified Reference Materials for Genetically Modified Organisms (GMOs)

The reference materials are produced either on IRMM's own initiative (e.g. to replace exhausted stocks of BCR reference materials) or on requests from research, public or industrial organisations and/or partners. To name but a few, examples of such partners are the International Federation of Clinical Chemistry (IFCC), the EC Directorate General Agriculture and Research (Standards, Measurements and Testing), the European Working Group on Reactor Dosimetry (EWGRD), the Istituto Superiore di Sanità (IT), Fluka Chemie AG (CH) and others.



Selection of Charpy Reference Materials for testing the impact toughness of steel

The production facility at IRMM has been designed over 4 decades largely by staff of the Institute and is now probably the most advanced and sophisticated of its kind world-wide. It can, for example, enable the production of large quantities of high quality biological and environmental candidate reference materials. Its unique multi-functional and flexible production laboratory contains clean chambers, cryogrinding, freeze drying, high purity milling and ultrafine classification and levitation melting machines. These facilities are presently being extended to the production of potentially infectious materials and/or materials where cross contamination through ambient air is critical (e.g. RMs for DNA analysis).

Adjacent to this facility is a highly modern analytical laboratory equipped with a.o. various spectroscopic methods, neutron activation analysis, gas and liquid chromatography (in the future various PCR and immunochemical techniques will be added) used for in-process control and certification analyses.

Reference material projects are funded by IRMM's specific programme, income from CRM sales, institutional support to the Commission, participation in shared cost actions and third party work. In 1999 more than 10 000 CRM samples were sold world-wide for a total value of ca. 1.2 MEUR.

FUTURE PROSPECTS

The need for CRMs in traditional fields like agriculture, environment and industry will continue to grow at a steady rate with accelerated growth expected in the fields of clinical medicine and biotechnology.

For example, IRMM will continue to provide reference materials for the different types of genetically Modified Organisms (GMOs) that are released and used - either directly or indirectly - for food production. Additionally, IRMM with its sister Institute for Health and Consumer Protection (IHCP), Ispra, will provide the authorities with the most suitable analytical methods for detecting GM crops and foods and thereby grant EU consumers the freedom to choose between GM and non-GM products. IRMM and IHCP have the expertise and means to create the first ever specimen bank for genetically modified materials, an idea that is presently under discussion. Such a specimen bank would not only provide an excellent service to the European citizen but also offer global standards for the detection of GMOs to all world nations. Many countries, including Australia, South Korea and Japan are now considering the mandatory labelling of all genetically modified products and in the USA, an intergovernmental panel to develop recommendations for the labelling of crops has been established.

Likewise, research will continue to combat both the BSE and dioxin crisis, new CRMs will be certified for clinical chemistry, exhausted CRMs will be renewed and IRMM will remain at the service of the EC always ready to quickly respond to the requests and the needs of the European citizen.



IRMM Storage Room (18°C) for Reference Materials



CONTACT

Dr. Jean Pauwels
Tel. +32 (0)14 571 722
Fax. +32 (0)14 590 406
jean.pauwels@irmm.jrc.be

MAJOR SCIENTIFIC RESULTS IN 1999

BSE Research - Evaluation of Post-mortem Testing

An important step forward was made in the fight against BSE (Decision 96/49/EEC). Co-ordinated by Directorate General Health and Consumer Protection and in collaboration with expert institutes, the IRMM prepared and dispatched over 14000 samples for evaluation of four candidate post-mortem BSE diagnostic tests. These tests produced some 5290 results that were evaluated by IRMM. The resulting statement from the Commission's scientific steering committee spoke for itself;

"Three of these tests can identify animals clinically affected with BSE".

This request required special laboratory facilities, needed to be completed within a 6 month time period and the results have been published in 'Nature'.

BSE Research - Proficiency Testing Samples

The production of ten Proficiency Testing (PT) materials for the "Detection of bovine mitochondrial DNA in animal feeding stuff of plant origin" was completed with 26 sets of samples dispatched to participants from expert laboratories in Europe.

Four proficiency testing materials for the "Determination of the appropriate heat treatment of animal meals" were prepared. From each composition, 195 samples were dispatched to IHCP, Ispra, Italy thereby completing this project.



Preparation of BSE samples in a specially designed glove box

GMO Research

In accordance to the EC Directive 258/97, IRMM has produced and certified the world's first CRMs for the detection of GMOs. In 1999, the production of GMO PT samples for quantitative Polymerase Chain Reaction (PCR) analysis were completed through the production of 1000 units each of 1 % and 5 % RR-Soya and Bt-176 Maize.

For Bt-11 maize, 3 CRMs were certified with 1000 units of 1 and 2 % being produced. Two evaluation tests (DNA/PCR and ELISA) were completed together with IHCP, Ispra and between 500 and 800 units each of 15 different processed food PT-samples containing GMO's were produced.



Samples of original corn produce

Response to Dioxin Crisis

With the advent of the 1999 dioxin crisis, sales of BCR reference materials for PCB analysis rose, in some cases, by several hundred percent. To provide further support an immediate action was undertaken by IRMM to prepare and certify (doped) pork RMs and to make this set of CRM's in pork fat matrix available for quality control purposes in laboratories EU-wide. A proficiency testing campaign of laboratories for PCB testing in pork fat, which have been analysing food for the presence of PCBs, will be organised and further RMs for PCBs in egg yolk powder, milk powder and (pork) meat are envisaged.

Participation in FP5 Shared Cost Actions

In accordance to the EC regulation 26/94 and as requested by CEN-TC 264 WG1, the preparation of dioxin standard solutions (10 solutions, 16110 ampoules), dioxins in sludge and dioxins in fly ash were completed this year. Moreover, 3 test-batches for trace elements in water (200 ampoules of 100 ml each) were prepared in 1999.

For the certification of a synthetic polymer reference material for multi-element analysis, the preparation, homogeneity control and certification analyses were completed in 1999, and the draft certificate and certification report were produced. In the frame of the IMMACO project, 320 sets of bronze samples (CRM 691) were produced and the five compositions were analysed by XRF, PIXE and NAA.

Finally the preparation of test materials for Cu and fibres in feed was completed with six animal feed test materials of pig and dairy feed being prepared and distributed for a preliminary ring-test.

CRMs for Pollution Control

In collaboration with the EC Directorate General for Research, 4 candidate reference materials were prepared for trace elements in sediments and fly ash in order to replace exhausted BCR CRMs. Homogeneity control studies for the 4 candidate reference materials were completed and the reports are being finalised.

On the topic of standard solutions, certification measurements for the purity of heptane and iso-octane reference fuels (IRMM CRMs 441 and 442) were completed during 1999. A certification report is in preparation.

Clinical Reference Materials

In collaboration with the Directorate General for Research and the International Federation for Clinical Chemistry (IFCC) and in accordance to EC Directive 98/79/EC, the certification of a serum panel for cortisol has been finalised. The certificate and report are available on <http://www.irmm.jrc.be> and the material is now available for sale.

The certification of multi-enzymes made significant progress with new Standard Operating Procedures (SOPs) being accepted by the IFCC working group. An intercomparison demonstrated good applicability of the new SOPs. The certifications of all 4 enzymes (ALAT, LD, GGT, CK-MB) at 37°C were completed and the certification documents are being finalised according to GUM.

For the certification of the enzyme ASAT, a first commutability study of two candidate materials was completed.



Clinical tests need to be verified using Certified Reference Materials

Management of BCR Certified Reference Materials

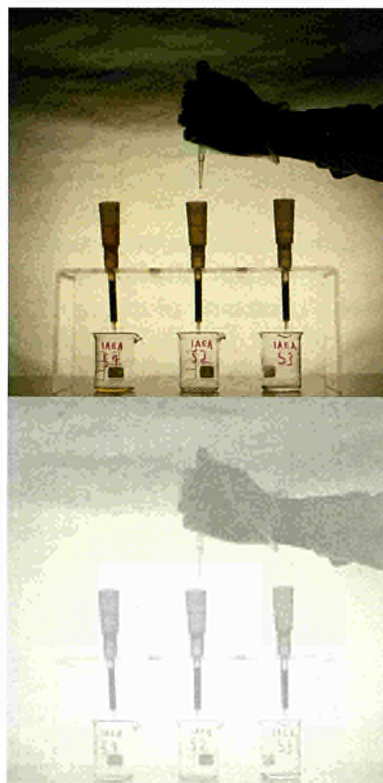
In 1999, a total of 8181 samples have been sold and distributed through 1998 processed orders. Stability monitorings were initiated, either internally (NAA, GCMS, etc) or externally, for the control of more than 100 existing BCR and/or assimilated IRMM reference materials. Furthermore, in accordance to EN 10045-2, IRMM certified 16 new CRM batches for charpy testing. It also participated successfully in an international evaluation of charpy reference specimens involving Japanese, French and American metrology institutes.

1463 units of whole milk powder to be certified for nutritional properties (in replacement of the exhausted BCR CRM 380) and 1486 samples of skimmed milk powder to be certified for fat content (BCR CRM 685, request of Directorate General Agriculture) were prepared. For the certification of sulphur and total glucosinolate in colza (replacement of existing BCR CRMs) all stability studies were completed.

Preparation and Characterisation of Nuclear Samples

In the IRMM sample preparation facility 322 accelerator/reactor samples were supplied and 67 units of neutron dosimetry CRMs were sold.

Among the targets prepared was a ^{26}Al sample made with yield disc to 100 % from a very limited quantity (1017 atoms) of high value starting material, and ^{149}Sm and ^{123}Te deposits prepared by "molecular plating" (a form of electrodeposition) for neutron cross-section studies. Alloys for reactor fluence rate monitoring were prepared by levitation techniques and low melting point entectic compositions were made and encapsulated for use and reactor temperature monitors. Plastic substrate foils for thin deposits were prepared by spin-coating.



Separation of actinoids from an environmental matrix using extraction chromatography

JRC CENTRE FOR CHEMICAL REFERENCE MEASUREMENTS

Today reference analytical measurements have become indispensable cornerstones for ensuring the safety of food, consumer products and a clean and indeed secure environment. With the expansion of the EU and an ever increasing importance placed on trade with the pre-accession countries, the USA, Asia, Australia, South America, etc., the need for global reference methods has never been so pronounced. To further augment this need, the European citizen demands higher quality as well as more information on the various consumer products that are offered on today's markets.

The European Commission plays a leading role in assuring and controlling the quality of consumer products. In the agriculture sector alone, the EU has built up a body of legislation on food safety and animal/plant health which are binding in all countries of the EU and which partially apply to non-EU countries exporting animals, animal products, plants and plant products to the EU.



Selection of hair dye forming compounds

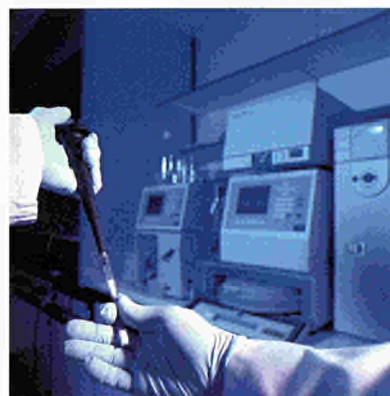
The Analytical Chemistry Unit at IRMM provides the scientific and technical support needed for the implementation of EU policies related to life and environmental science and consumer protection. Its highly experienced staff, unique facilities and flexible approach deems it ideal to respond to urgent requests by the various DGs.

This unit co-ordinates a spectrum of research activities ranging from the detection of antibiotics in domestic meat supplies, identifying substances in cosmetic products listed in the Cosmetic Directive and detecting the presence of radionuclides/trace elements in food to the speciation of selenium in feed stuffs and the development of a reference method for determining sugars in industrial syrups.

A database for the safety of consumer products is also being created wherein the associated risks and respective expert/help institutes will be made available to consumers with the touch of a button. This database has already been installed on the internal IRMM network and will soon be accessible via the World Wide Web.

The metrological service provided by the Analytical Chemistry Unit is the backbone to all IRMM activities. Precision mass determinations provide direct traceability to the SI unit of mass, and form the underlying traceability link in amount of substance metrology to the SI unit - the mole. Other measurement capabilities of the laboratory include density, length, thickness, surface area and the alignment of beam tubes and flight paths.

The analytical equipment laboratories at IRMM have been carefully designed over a 4 decade time period. It hosts chemical and radiochemical laboratories with high performance analytical instrumentation including ICP-OES, ICP-MS, AAS, LC-DAD, LC-ED, LC-MS-MS, CZE-DAD, SFE, XRF, UV-Vis spectrometry, electrochemical techniques, α and γ spectrometry, PIXE, RBS and metrology instrumentation.



*Determination of milk adulteration
by HPLC coupled to Diode Array
Detection (DAD)*

FUTURE PROSPECTS

The provision of scientific support for implementing EU policies related to consumer protection and to life and environmental sciences will require new reference methods to ensure measurement results of true provenance. This is particularly true in light of the relatively recent but fast growing awareness of metrology in chemistry especially in terms of the traceability and uncertainty of chemical measurements. Furthermore the booming field of biotechnology will require scientific benchmarking and control - only achievable through the use of certified reference measurements and reference materials.

Future activities dealing with food analysis, life sciences and consumer protection will be expanded. Many contacts with national institutes, universities and industry have already been established in order to create different networks in the field of antibiotics, speciation and cosmetic model formulations.

A special focus for the unit will be to further maintain and strengthen relations with the different Directorate Generals and thereby fulfill the JRC role in providing scientific and technical support towards the implementation of EU policies.



CONTACT

Prof. Dr. Dr. Adela Rodriguez
Tel. +32 (0)14 571 200
Fax. +32 (0)14 584 273
adela.rodriquez@irmm.jrc.be



Magnus Johansson (SE) and Beatriz De La Calle (ES) working on the separation of selenium species in food products

MAJOR SCIENTIFIC RESULTS IN 1999

Support to Legislation on Cosmetic Products

In 1999, IRMM maintained the chairmanship of, and provided support to, the working group "Methods of chemical analysis for Cosmetics" as well as participating in several subgroups.

In the laboratory, the development of a candidate European reference method for oxidative hair dye analysis continued in accordance with the Cosmetic Directive (76/768 EEC, 93/35 EEC) and its sixth amendment. Hair dye matrix interference studies were completed and five artificial liquid formulations were accurately quantified. Additionally, quantitative determination of seven hair dyes in five different commercial products from L'Oréal and Garnier (dark and light brown, dark and light blond) was performed.

Discussions on cosmetic model formulations for analytical purposes have also progressed significantly.



Range of cosmetic formulations

Selenium Speciation in Agricultural Feeding Stuffs

Selenium is both essential (humans require >50 µg/day) and toxic (max. 200 µg/day) and this narrow concentration difference demands a better understanding of this element's speciation in order to ensure the protection of human health. In collaboration with DG Agriculture and in support to the EC Directives 74/63/EEC and 87/153/EEC, a hyphenated analytical method (IC-MW-HGAAS) was developed for selenium and all separation interferences were carefully studied. The separation of four Se species was achieved in a standard solution and this new method was applied to a reference material of white clover (CRM 402), certified only in total Se.

IRMM also participated in an interlaboratory comparison (9 participants) for selenium in a selenium enriched yeast material. This intercomparison was carried out in the frame of the "Speciation 21" thematic network. The determination of the total seleni-

um concentration was finalised in 1999 and the identification and determination of several Se species in yeast by IC-MW-HGAAS is presently in progress.

Sugars in Industrial Syrups

This project aims at replacing the Lane-Egmon method which is presently being used as the official method but is now obsolete, by a more modern method based on high performance liquid chromatography separations. This work supports the EC Directive 79/796/CEE, the EC Regulation 394/70 and is carried out in collaboration with DG Agriculture. In 1999 a HPLC method (without derivatisation) using a refractive index detector was developed and tested using artificial BCR reference materials. The method yielded good agreement with the reference values. A second HPLC method with post-column derivatisation and UV detection was developed and very successfully tested using reference materials 351J and 351K. Both methods give similar and reproducible results. The report is being finalised for DG Agriculture.

Proteins for Milk Authentication

Milk has a variable protein composition depending on its origin (cow, sheep, goat, etc) and/or treatment. The precise protein characterisation of each type of milk can enable the detection of fraudulent manipulations and for this reason a new project commenced in 1999 in collaboration with Agriculture and Health and Consumer Protection DGs and in support to the EC Directives 83/417/CEE and 91/32/CEE.

Studies on milk reference materials (CRM 063R BCR and 1549 NIST) were carried out through the separation of 11 components via RP-HPLC-DAD. From the 11 components, 8 have already been identified as the caseins (K-CN, α_{S1} -Cn, α_{S2} -Cn and β -CN) and whey proteins (β -Lg B/A and α -La). Preliminary quantification of the major milk proteins in several commercial milks (pasteurised, UHT, milk powder) with RP-HPLC (C8 column) has also been carried out.

Antibiotics in Domestic Meat Supplies

The problem of antibiotics in domestic meats has prompted intervention by the EC. This new project, in support to EC Directive 70/524/CEE and EC Regulation 2821/98, commenced with literature research and a new collaboration with the University

of Liège (Department of Veterinary Medicine). Two families of antibiotics (macrolides and quinolones) were selected and commercially available standards were acquired. The separation of 5 quinolone antibiotics by CZE-DAD was carried out successfully and the same compounds were submitted for ESI-MS/MS fragmentation studies. Excellent results with a low limit of detection was achieved.

New Analytical Laboratory for Food Safety

In order to accommodate the ever increasing requests for reference methods for food safety, a new laboratory for LC-MS-MS was set up and first tests were successfully carried out with several metalloproteins.

To apply the method, a screening of several pesticides in oranges commenced and is presently ongoing.



Gery Van Vyncht (BE) determining antibiotics using hyphenated HPLC-ESI/MS/MS

Metal-binding Proteins

In order to foster the development of new methods, samples of proteins and metal-binding proteins were selected (milk, wheat) and the total metal concentration (Cu, Fe, Zn) was determined in a milk reference material. Additionally, preliminary studies were carried out for protein separations in milk using size exclusion chromatography in order to allow the determination of associated metal ions. On a different topic, electrochemical characterisations of human foetal liver and rat liver metallothioneins and of α and β MT peptidic domains were also successfully carried out. In the last case, results have been confirmed using ESI-MS. The separation and characterisation of MT isoforms using CZE-DAD has continued and has made progress, while the application of direct ESI-MS and LC-ESI-MS has allowed the separation and identification of non-acetylated forms.

Metallothionein Workshop

The COST project "Chemistry of Metals in Medicine" has been accepted and the first meeting with 5 partners took place during the 2nd International Workshop on Metallothioneins organised by IRMM in October 1999. This successful workshop (1st Euroconference) was attended by about 100 people from 21 countries and as a result a special and dedicated issue of "Analysis" (European Journal of Analytical Chemistry) will be released in 2000.

Radionuclides in Food

The enlargement of the EU has prompted an increasing number of requests from national and international bodies to certify new and/or improve existing food reference materials needed for the analysis of radionuclides (Council Directive 96/29/EURATOM of 13th May 1996).

At IRMM a reference method to determine trace nuclear impurities in food materials is being developed using ICP-MS. Matrix effects, column capacity and elution profiles have been examined closely and a new separation method based upon extraction chromatography has been developed. Alpha spectrometry analysis and a detailed study of the sorption selectivities of Th, U, Pu and Am from water have also been conducted.

The Thorium Network

The European Directive (EURATOM 80/836) laying down the basic safety standards for the health protection of the general public and workers requires that environmental and manufacturing materials exceeding 100 Bq/g Thorium must be reportable. The Directive has been changed and in the near future this value will have to decrease. However, it has been shown by the National Physical Laboratory (UK) that there is a real need for reference materials and validated methods for thorium analysis. For this reason a network has been set up by Directorate General Research and IRMM is participating as expert laboratory.

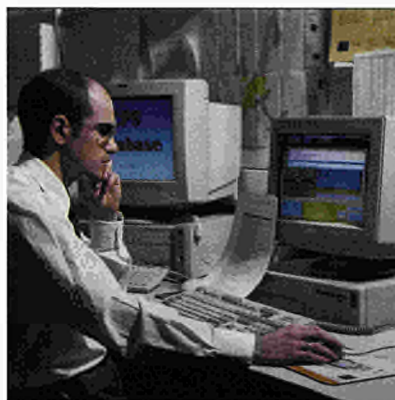
In this context, as a first investigation into the performance of different methods, concentrations of two solutions supplied by NPL to 14 laboratories as an intercomparison exercise were determined at IRMM using ICP-MS and alpha spectrometry and the results of both methods were in excellent agreement with NPL values.

Ion Beam Reference Materials and IMMACO

A Sb implanted into Si RM was prepared and the internal Au standard was evaporated onto the implanted chips. Uniformity measurements of Rh as internal standard on Bi implant in Si was completed and evaluated, uniformity and certification measurements of Sb implant in Si with Au as internal standard was also completed.

For micro-structured RMs the measurements with X-ray microbeams to develop procedures for halo determination were completed. Also length metrology of individual chips was performed through calibration with the photomask linewidth standard.

IMMACO (Copper alloys) PIXE measurements to support the certification were completed, the report was written and all XRF homogeneity measurements were completed.



Javier Alba-Sanchez (ES) locating an expert institute that will investigate a potentially dangerous product

Database for Consumer Protection

In collaboration with Directorate General Health and Consumer Protection and in accordance with the Directive 92/59 EEC, IRMM is implementing a database for consumer product safety. In 1999, a new design for the database user interface and infrastructure has been put forward, as well as subsequent steps to deploy the database on the web. The necessary tools for the new design have been purchased, the classification of consumer products and hazards was finished, a prototype database was created, migration of data was completed and the relationships between data have been re-established. This improved system now contains less data than the former, but in a much more efficient arrangement. The prototype database is presently on trial and the final product will shortly be deployed onto the World Wide Web.

JRC REFERENCE LABORATORY FOR ISOTOPIC MEASUREMENTS

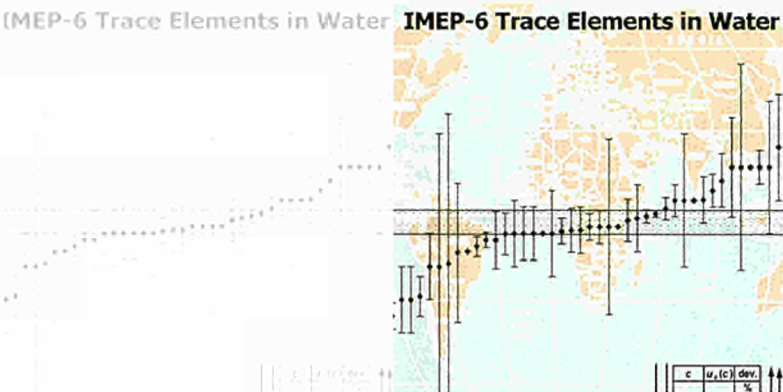


JRC REFERENCE LABORATORY FOR ISOTOPIC MEASUREMENTS

Chemical measurements form the basis of important economic, political, environmental, medical and legal decisions. Each day millions of measurements (10 billion data per year) are carried out throughout the world, the results of which will, for example, determine whether or not the food we eat is safe, the air we breathe is clean and the earth we live of is contamination free.

The real basis for decision-making and implementing regulations depends on the comparability and reliability of such chemical measurements. This requires that measurements should be traceable to "stated references" and where possible to the International System of Units (SI - *Système International d'Unité*). For chemical measurements of amount-of-substance, the base unit is the mole.

What often happens with measurement results is that a mean or "consensus" value is taken as the closest to the true value. Accreditation schemes help ensure that measurements conform to formal procedures but do not fully tackle the problem of the metrological reliability and comparability of the results. This explains why the traceability of chemical measurements is often lacking.

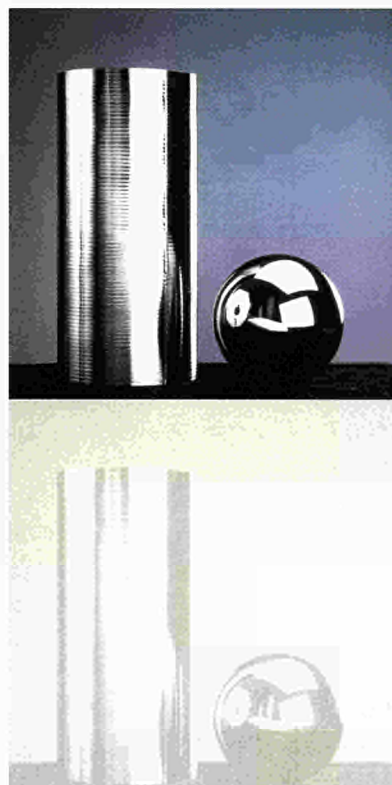


A typical spread of analytical results from an IMEP round

In 1993, these matters triggered the International Bureau of Weights and Measures (BIPM, Paris) to set up a Consultative Committee for Amount of Substance (CCQM), of which major National Metrology Institutes (NMIs) and IRMM (as the Transnational Measurement Institute (TMI) of the European Commission) are members. The CCQM deals with matters of traceability of chemical measurements on a global scale. At the European level EUROMET, the association of European NMIs including IRMM, is the competent regional body.

It also operates a Committee on Amount-of-Substance (AoS) Measurements.

There also exists a global network of NMIs including the major trade regions, like EU, USA, Japan and the Asia-Pacific Region between whom international measurement equivalence is presently being established through a system of "key comparisons" (KCs). Such KCs, which are organised by BIPM/CCQM on a global basis and by EUROMET for the European region (including EU, associated and accession states), have provided and will continue to provide information on the degree of equivalence of analytical data obtained in NMIs. This system intends to support the Mutual Recognition Arrangement (MRA), which was constructed to establish equivalence of standards and measurement certificates. On April 14th, 1999 the official representation of the European Commission in EUROMET was transferred to the IRMM. Moreover, on October 14th 1999, IRMM together with the Directors of Member State National Measurement Institutes, the IAEA and the BIPM, signed the Mutual Recognition Arrangement in Paris. BIPM explicitly invited IRMM (on behalf of the EC) to sign the MRA. IRMM's involvement is based on its proven measurement capability in primary isotopic and elemental measurements.



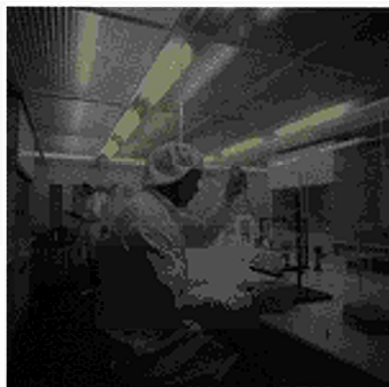
Silicon spheres prepared at CSIRO (Australia) and used in the international Avogadro projects'

With the largest and probably most modern isotope mass spectrometry laboratory in Europe complemented by an adjoining class 10 ultra clean chemical laboratory, the scientists at IRMM have been producing SI-traceable values for almost 4 decades now. Primary isotopic gas standards, isotopic reference materials, spike isotopic reference materials are produced and isotope dilution is applied as a primary method of measurement for measuring the elemental amount content in various matrices.

Sales of Isotopic RMs mounted to 84,480 EUR in 1999 and the complete list of IRMs can be accessed under <http://www.irmm.jrc.be/mrm.html>

For nuclear Isotopic RMs (IRMs), the IRMM will become the main supplier to Japan, since the IAEA have now stopped supplying JNC (Japanese Nuclear Cycle Development Institute, Tokai) Japan.

This level of excellence at the JRC Reference Laboratory for Isotopic Measurements has brought to bare on renowned projects like the International Measurement Evaluation Programme (IMEP), the Avogadro project, the world's first Primary Isotopic Gas Standards (PIGS) and the capacity and established impartiality to perform "referee" measurements needed for Nuclear Safeguards and to maintain and expand an independent European metrological capability in the area of nuclear analysis.



*Ultra Clean Chemical Laboratory
with Michael Berglund (SE) and
Carmel Hennessy (IE)*

FUTURE PROSPECTS

The application of metrology in chemistry, still in its infancy, will undoubtedly flourish and grow over the coming years and IRMM is at the heart of this change in thinking and realisation. Its tools, in the form of International Measurement Evaluation Programmes, primary isotopic measurements and its undisputed capability in the area of nuclear analysis, will pave the way towards generating an internationally structured system and thereby help achieve the ultimate aim - widespread international equivalence of chemical measurements.



CONTACT

Dr. Philip Taylor
Tel. +32 (0)14 571 605
Fax. +32 (0)14 571 863
philip.taylor@irmm.jrc.be

MAJOR SCIENTIFIC RESULTS IN 1999

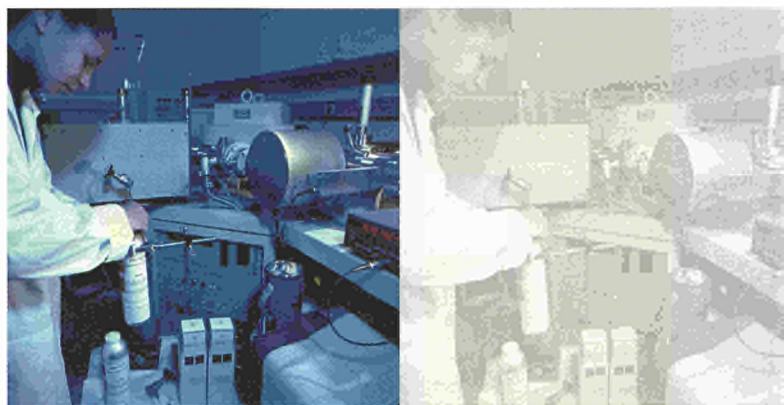
First Global Key Comparison using IMEP-9 Samples

In 1999, the BIPM Key Comparison (BIPM-KC) in the area of chemical measurements has been successfully completed with IRMM as the pilot laboratory. For this, IMEP-9 samples (trace elements in water) were made available to 10 different National Measurement Institutes from 4 different continents. Simultaneously, the same IMEP-9 samples were provided to 200 participant laboratories in the water analysis sector (from 35 different countries) and the element amount content was measured. Additionally, IMEP-9 was chosen as a pilot project for European Accreditation (EA), who used it to enable result oriented evaluation of performance.

It provided an objective picture of the degree of international measurement equivalence between the NMIs of Germany, France, Great Britain, the EC, Russia, USA, Japan, Korea and China. Furthermore, for the first time ever it was possible to view measurement quality across laboratories which have different metrological roles. As expected, results obtained in field laboratories showed a larger scatter. It thus clearly demonstrated the value of IRMM's IMEP approach and the role IRMM can play in this context.

Primary Isotopic Gas Standards (PIGS)

PIGS enable SI traceable isotope ratios to be obtained in areas of food, clinical, atmospheric and environmental chemistry. Excellent progress was achieved in 1999 with the completion of the SF_6 certificate, the preparation of a carbon PIGS (CF_4) which was bottled at Messer Griesheim GmbH, Germany and CF_4 PIGS were measured on the "Avogadro II amount comparator". Additionally, SI-traceable ratios of carbon and oxygen were measured on pure CO_2 , also prepared in cooperation with Messer Griesheim GmbH. Methods were successfully developed to convert CO_2 into C and thereafter further fluorination to CF_4 was achieved. Application of this method to natural products showed that beet, cane and maple sugar were successfully converted into CF_4 with very good chemical purity and the $^{13}\text{C}/^{12}\text{C}$ results obtained were in good agreement with the literature values.



Yetunde Aregbe (AT) performing the highest quality isotopic measurements with IRMM's Avogadro II Mass Spectrometer

Avogadro Project

This work contributes towards the three highly prestigious international Avogadro projects which aim at redefining the kilogram. IRMM is the only laboratory in the world that can supply molar mass values of such high quality. In 1999, new differential isotopic measurements for SiF_4 (carried out in cooperation with the Institute for Mineral Deposits, Beijing) demonstrated and confirmed that the original (1994) IRMM molar mass measurements on the anomalous Japanese Shin-Etsu material were excellent. 40 samples from different Avogadro projects (PTB-Germany, IMGC-Italy, and NRLM-Japan) were converted into SiF_4 by fluorination. The samples were then measured on a differential gas mass spectrometer and on the IRMM Avogadro-II Amount Comparator (absolute isotope ratios). The results further confirmed a linear relationship between molar mass and density of better than 10^{-6} .

Production and Certification of Isotopic Reference Materials (IRMs)

The Fe spike IRM (1000 mg/ml solution) was prepared, ampouled, and the certificate has been finalised (IRMM-634). For the ^{50}Cr IRM and $^{\text{nat}}\text{Cr}$ spikes, three 1000 mg/ml solutions were prepared and ampouled, measurements were finalised and the certificates were written.

Solutions for the Boron IRM were prepared and measured and the ^{202}Hg spike and Cl spike IRMs were prepared and ampouled.

Additionally, two new tasks were launched for S materials with different isotopic compositions and Zn spike IRMs. For S, a total of 325 ampoules have been filled.

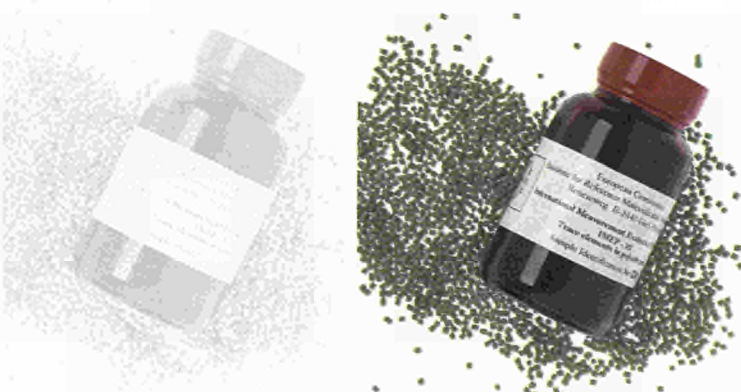
IMEP Rounds

IMEP-8 (carbon/oxygen isotope ratios in CO_2), and IMEP-11 (metals in car exhaust catalyst) were both chosen as pilot projects for EA, and for both rounds data were received and processed in 1999. IMEP-8 involved 27 laboratories from 14 countries whilst IMEP-11 involved 36 labs from 16 countries. At short notice and on request of EA, IMEP-13 was initiated (trace elements in polyethylene) involving 85 laboratories from 25 countries. Samples were distributed, results collected and evaluated and the participant's report will be submitted in April 2000. In the context of the CCQM, IMEP-14 (trace elements in sediment) was launched. Samples have been distributed to 240 laboratories and the collection of results has already commenced. Most of the IMEP rounds are and will be registered as EUROMET projects and are proposed as EURACHEM-EUROLAB-EUROMET proficiency testing projects.

New and Future IMEP Rounds

IMEP-12 (trace elements in water) was initiated in support of Directive 98/83/EC. Additionally interactions with IMEP customers led to plans for the following new rounds:

- > IMEP-17 (trace and minor constituents in serum) for the clinical sector
- > IMEP-18 (sulfur in fuel) for industry
- > IMEP-19 (Cd in rice) for the food sector



IMEP-10 bottle for the determination of Cd, Cr, Hg, Pb, As, Cl, Br, and S in polyethylene

Safeguards - IRMs and Spike IRMs

A natural uranium spike material (IRMM-056) was prepared and certified in the frame of a one month training programme for TACIS (Bochvar Metrology Laboratory, Russia).

A new ^{240}Pu spike (IRMM-083) was prepared and certified and a ^{235}U spike (IRMM-054) was ampouled. Stability verification measurements were carried out on mixed U/Pu solutions and a new solution (U250/Pu2.5) was prepared, ampouled and certified. The preparation of new ^{242}Pu and ^{244}Pu environmental spikes for environmental applications got underway and the ampouling for both spikes was completed. Finally certificates were completed for a natural uranium spike material (IRMM-054) and for a ^{235}U spike material (IRMM-056).



Thomas Prohaska (AT) measuring isotope ratios on a multiple collector ICP mass spectrometer

Safeguards - Uranium Isotopes

In collaboration with the official USA Safeguards Laboratory in New Brunswick, USA, IRMM is engaged in the production of a set of global isotopic reference materials for the nuclear accounting of uranium.

In 1999, a series of UF_6 starting materials were purchased and characterised for future mixing requests. A series of uranium oxides with certified isotopic abundances were produced. These will be used for the production of U-doped glasses (ground to particle sizes of around 10 μm) for the IAEA support programme. Such IRMs will be the first ever of their kind and will be used as certified test samples. Their uranium isotopic composition will be measured by laboratories in the field of nuclear environmental safeguards.

Uranium minor isotope abundances were measured on the same samples. Finally the isotopic abundances for a set of 4 uranium materials (UF_6) were

calculated for a REIMEP-15 campaign (Regular European Interlaboratory Measurement Evaluation Programme) and these samples are ready to be dispatched to participants.

Safeguards - Launch of NUSIMEP Programme

The Nuclear Signatures International Measurement Evaluation Programme (NUSIMEP) forms the basis to achieve measurement equivalence in nuclear trace analysis of environmental samples. NUSIMEP-1 (completed in 1998) was a series of diluted U and Pu isotopic reference materials produced for the IAEA, and based on the success of these RMs, new series have been planned and will be offered for general use. NUSIMEP-2 will be a series of 4 near-natural uranium samples, about 10ppb in dilute nitric acid, suitable for testing and proving techniques for measuring isotope abundances of uranium in environmental samples. On July 23rd 1999, NUSIMEP-2 was launched and the execution of this programme is presently in progress.

Safeguards - General Activities

For external quality control work, over 20 laboratories have responded positively to a forthcoming REIMEP round.

For environmental RMs, and in collaboration with the EURATOM Inspectorate, Luxembourg, blank natural uranium contents in a series of (unused) swipes were measured by IDMS and the isotopic ratios were measured by ICP-MS with good sensitivity and agreement. The results were submitted to Luxembourg.

Also for environmental safeguards and in collaboration with the International Atomic Energy Agency (IAEA), 1 kg of base glass has been ground and the blank level of uranium in this glass was measured at Los Alamos National Laboratory (LANL), USA.

Denuclearisation of IRMM

A list of materials for disposal was drawn up and packaging of materials to be dispatched to the Studie Centrum voor Kernenergie (SCK), Belgium has been completed. 23 tin cans of 1.5 L each have been packed with 1741.7425g plutonium and 493.916g uranium.

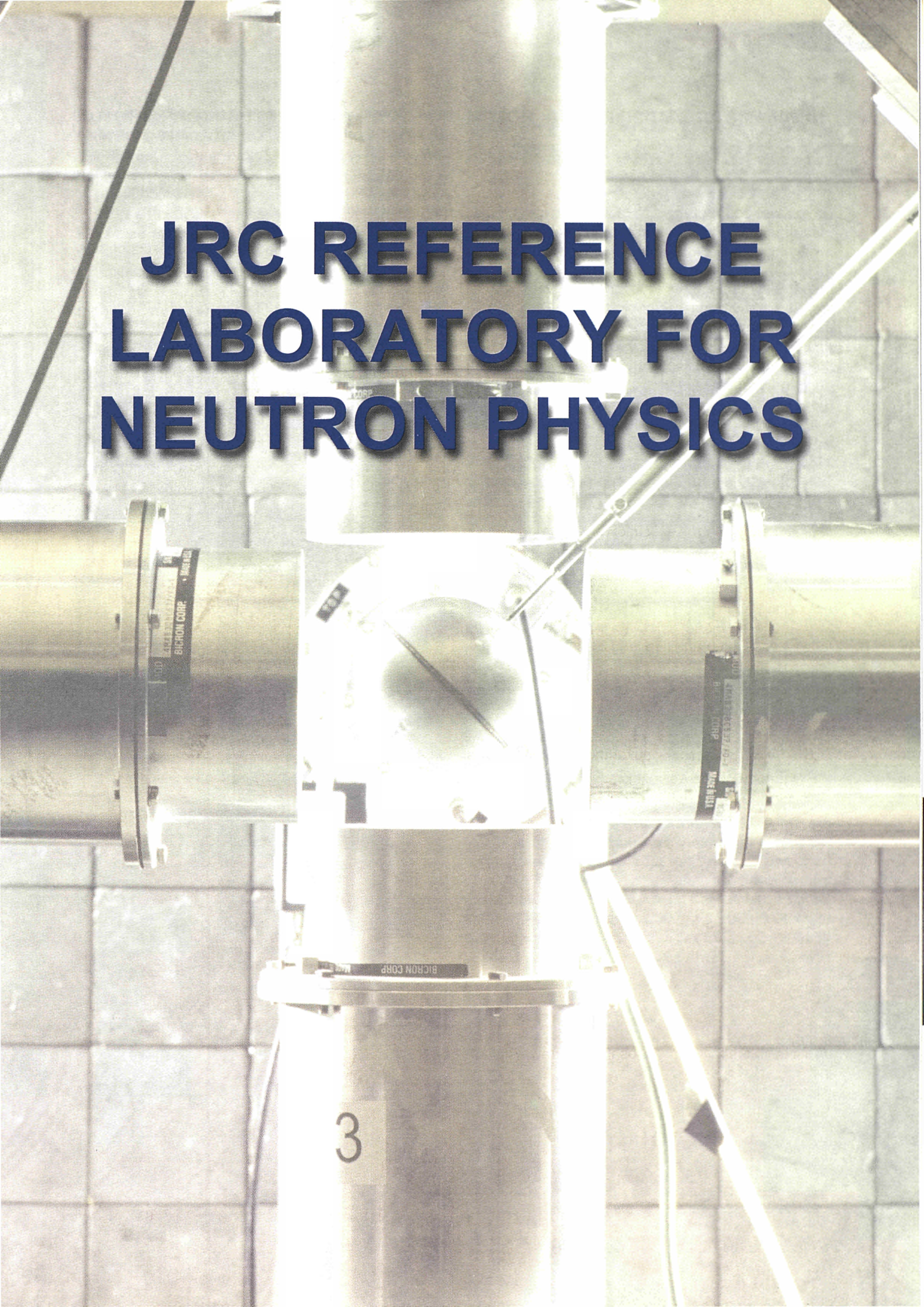
Two lightly contaminated gas mass-spectrometers have been dismantled whereby the contaminated

parts will be sent to ESO (Euratom Safeguards Office) Luxembourg. Additionally, 7 glove boxes were transported to the waste storage room. Many "small" laboratory devices were cleaned, measured and decommissioned and three laboratories are now ready for refurbishing. 7 glove boxes were decommissioned and approx. 32m of glove box extraction ducts were dismantled, measured and disposed of.



Heinz Kuhn (DE) measuring nuclear materials on a thermal ionisation mass spectrometer

JRC REFERENCE LABORATORY FOR NEUTRON PHYSICS



3

JRC REFERENCE LABORATORY FOR NEUTRON PHYSICS

Considering that one third of the total electricity produced in western Europe comes from fission reactors there is a fundamental responsibility to ensure and improve the safety of nuclear reactor installations, to advance technology for waste treatment and to protect the environment. One key way towards achieving this is through studying the interaction of neutrons with matter, a unique technology that also serves to advance research in material and medical sciences.

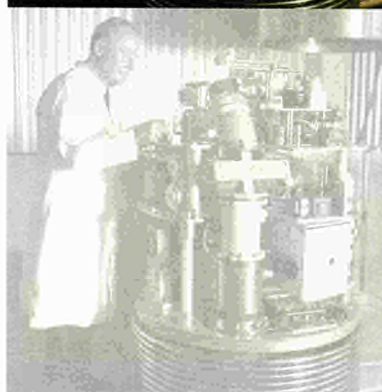
A network has been established to ensure that scientists and engineers world-wide have access to such valuable neutron data. This database is being maintained by two internationally renowned organisations, namely the OECD (Organisation for Economic Co-operation and Development) and the JEFF (Joint European Fission and Fusion File). IRMM is one of the major contributors to this network and additionally it conducts safety assessments of recent developments in this energy sector (e.g. increased fuel burn-up, plutonium recycling and waste transmutation). The neutron data measurement programme at IRMM is primarily determined by this network and in particular addresses the data needs formulated in the high priority request list continually issued and updated by the OECD. This request list is the major deciding factor for the different neutron data projects at IRMM which are described in the forthcoming pages.

Such specialised neutron interaction studies can only be carried out using unique facilities, of which there are only a few available world-wide. The JRC Reference Laboratory for Neutron Physics at IRMM hosts two such facilities with its 150 MeV white spectrum linear accelerator (GELINA) and 7MV Van de Graaff accelerator. These facilities have been maintained, upgraded and fine tuned for over 35 years and the scientific and technical staff has, over this

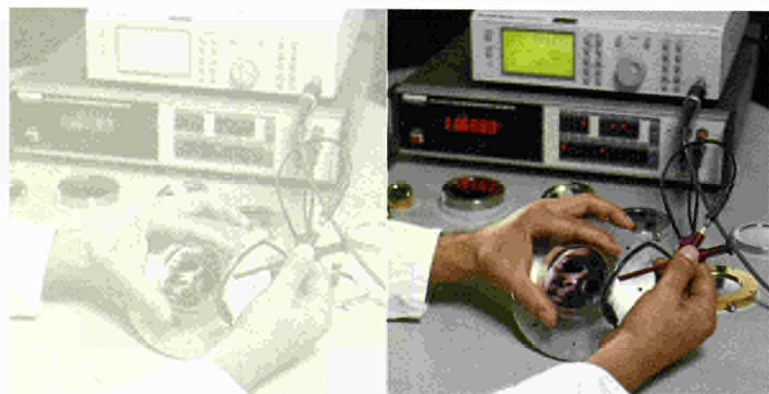
time period, acquired an extremely high level of expertise and competence in neutron measurements.

Both neutron sources (150 MeV white spectrum linear accelerator and 7MV Van de Graaff accelerator) were each kept operational in 1999 for ~2400 hours and 1990 hours respectively. Some 7 Mbytes of data were generated for neutron data work and submitted to the OECD. Neutron data measurements in the OECD area are co-ordinated by IRMM in the frame of the OECD Working Party on International Data Measurement Activities.

The financing of the research activities stems mainly from the institutional budget. The remainder is acquired via international scientific collaborations that require know-how, information and the use of measurement equipment.



Albert Crametz (FR) preparing the high voltage terminal of the 7MV Van de Graaff accelerator



Oliver Serot (FR) testing the functioning of the surface barrier detectors used in the charged particle detector.

FUTURE PROSPECTS

The future programme of the Neutron Physics Laboratory will evolve according to applications requirements in reactor and fuel cycle physics, especially with respect to safety aspects. The main emphasis will be on the measurement of data needed for safety issues related to increased fuel burn-up and plutonium recycling, and for feasibility studies of waste transmutation strategies, accelerator driven systems, and the Th fuel cycle. The detailed measurement program will further be guided by the High Priority Request List of the OECD. A closer collaboration with industry will be sought, especially for the definition of priorities assigned to the requests in the High Priority Request List. This list presently contains too many requests to be dealt with by the limited resources available world-wide in the neutron data measurement area.



CONTACT

Dr. Hermann Weigmann
Tel. +32 (0)14 571 475
Fax. +32 (0)14 571 862
hermann.weigmann@irmm.jrc.be

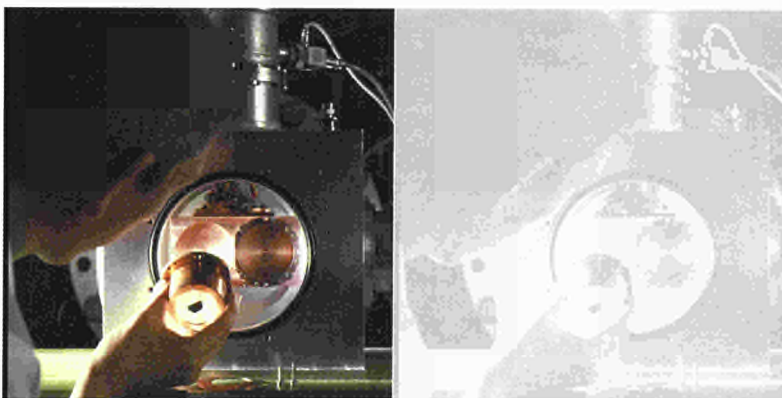
MAJOR SCIENTIFIC RESULTS IN 1999

Doppler Broadening at Low and High Temperatures

These investigations are important for the reactor temperature coefficient of reactivity, a prime safety parameter of nuclear reactors.

Progress in low temperature work was achieved through completing measurements on U(metal) and UO_2 at 14-300K. An improved treatment of Doppler Broadening based on the harmonic crystal model (DOPUSH) was implemented in REFIT. Experimental measurement on NpO_2 and Hg_2Cl_2 were also completed in 1999. First measurements for Hf(metal) were started.

For UO_2 high temperature work the furnace was built, tested and the pyrometer was calibrated. The furnace works well except above 2500°C where material problems arise. This material degradation problem is presently being investigated.



Peter Siegler (DE) mounting a UO_2 mono-crystal in the cryostat for the Doppler broadening experiment

Charged-Particle Emission Cross Sections

Neutron induced charged particle emission and gas production are the main mechanisms of radiation damage. Therefore, the knowledge of the respective cross sections and action characteristics (energy distributions of the emitted particles) is essential for the estimate of radiation damage to structural components of reactors or transmutation facilities as well as for dosimetry in radiation therapy and radiation protection applications. A feasibility study for a new project on (n,α) , (n,p) measurements got underway and it seems likely that a gas-TPC (time-projection chamber) will be used. A new data acquisition system was ordered and the design of a new experimental chamber is already under way. Measurements will commence in 2000.

Activation Cross Sections for Short-lived Products

Activation cross sections for short-lived products are important for the evaluation of reactor and radiation safety. Cross sections for 30 short-lived products were measured between 16 and 21 MeV at IRMM and for ^{99}Tc between 9 and 12 MeV at the FZ Jülich. This is a second measurement campaign in collaboration with Argonne National Laboratory on several isotopes of Cr, Cu, Mo, Se, Sr, Zn, Zr, V, and Tc.

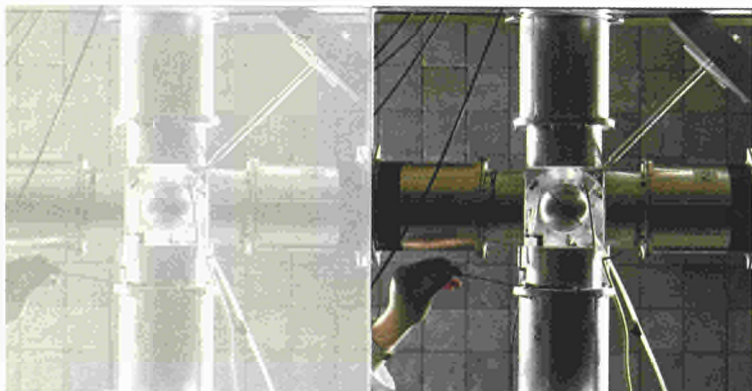
Reference Measurements on Actinides

The Thorium fuel cycle in connection with accelerator driven systems is subject of active research in EU Member States as it offers the possibilities of long-term nuclear energy generation, due to the large supplies of thorium, and the considerable advantages in terms of nuclear waste generation.

For $^{232}\text{Th}(n,\gamma)$ by activation, experiments are under preparation at the Van de Graaff and three samples have already been irradiated at 1 to 3.5 MeV.

For $^{232}\text{Th}(n,\gamma)$ by γ -detection, the experiments at Forschungszentrum Karlsruhe were completed and the measurements at the 150 MeV accelerator were finished.

For $^{234}\text{U}(n,f)$, thermal cross sections were measured at ILL (Institut Laue-Langevin), Grenoble (FR) and the fission yield measurements commenced. Cross section measurements in the eV to keV energy range also started at IRMM. The GELINA experiments in the meV to eV energy range confirmed the $1/v$ energy dependence.



^{84}Kr sample for neutron capture measurements at the 60 m flight path at GELINA

Cross Sections for Neutron Absorbing Materials

Being not only important for nuclear astrophysics, ^{84}Kr and ^{86}Kr are fission products with high yield,

making their cross sections an important factor for the physics of nuclear reactors.

$^{84,86}\text{Kr}(n,\gamma)$: $^{84}\text{Kr}(n,\gamma)$ was measured up to 200 keV and σ_{tot} -measurements started as well as the $^{86}\text{Kr}(n,\gamma)$ measurements. The ^{84}Kr transmission experiments were finished and the resonance parameters have been published. For the new Xe sample the transmission measurements have been completed and the analysis started. Also, an ^{82}Kr sample arrived and the first test experiments have been carried out.

Representing another neutron absorber, the $^{107,109}\text{Ag}(n,\gamma)$ measurements were also completed in 1999 and the data analysis is ongoing.

Feasibility Studies of Waste Incineration Projects

Representing a major environmental concern, long-lived fission products and actinides present in nuclear waste are under investigation for a possible transmutation into short-lived or stable products by means of neutron irradiation.

For $^{99}\text{Tc}(n,T)$, (n,γ) cross sections, the analysis of transmission data was completed (E-range: thermal to 100 keV) and published. The resonance parameters (up to 10 keV) will be sent to the NEA Data Bank.

For $^{237}\text{Np}(n,T)$, (n,γ) cross sections, (n,γ) measurements have been completed and the data analysis is ongoing.

For $^{129}\text{I}(n,T)$, (n,γ) cross sections, a 200 l solution containing ^{129}I arrived at IRMM and the apparatus for the separation of PbI_2 from the solution was prepared. A method was developed for high yield production of PbI_2 .

Data for Accelerator Driven Systems (ADS)

Accelerator driven systems are considered a major option for waste transmutation facilities. For their feasibility study, neutron data for target and construction materials of such systems must be known.

For $^{207,208}\text{Pb}$ and $\text{Bi}(n,\gamma)$ cross sections, measurements were completed, results published and sent to the NEA Data Bank.

For $^{208}\text{Pb}(n,n')$ cross sections, the main part of the measurements as well as normalization with a Ge-detector were completed at GELINA.

Finally for activation cross sections leading to long-lived products, a feasibility study is ongoing for $^{14}\text{N}(n,p)$, $^{51}\text{V}(n,n'\alpha)$, $^{94}\text{Mo}(n,p)$, $^{204}\text{Pb}(n,p)$. Irradiation of ^{94}Mo and ^{204}Pb was performed at the Van de Graaff and chemical separation and sample preparation work followed with 1/3 of the ^{94}Mo sample already separated.

Data on Inelastic Scattering

Inelastic scattering is the main mechanism by which neutrons lose energy in fast neutron spectrum systems. Therefore, inelastic scattering cross sections of structural materials (Fe, Al) have a decisive effect on criticality, those of coolant materials (Na, Pb) have a decisive effect on the coolant void effect, the most important reactor safety parameter.

The analysis of data with high neutron energy resolution was completed for the $^{56}\text{Fe}(n,n')$ cross section measurement. An additional experiment with a Ge-detector for normalization purposes was completed at the GELINA facility and the data were sent to Commissariat à l'Energie Atomique (CEA), Cadarache, France for analysis. Additional measurements and analysis are ongoing.

Data analysis for $^{238}\text{U}(n,n')$ was finalized. A Ph.D. thesis was presented and a publication is in preparation.

For $\text{Al}(n,n')$ cross section measurements, a simultaneous R-matrix fit of total and (n,n') cross sections is being performed, but work is ongoing for improvement. The analysis of total and inelastic cross sections was completed in 1999.



Hydrogen plasma in the radio-frequency ion source to produce the proton beam in the high voltage terminal of the 7MV Van de Graaff accelerator

The $^{10}\text{B}(n,\alpha)$ Standard

The ^{10}B standards have recently attracted attention due to discrepancies between measurements and the evaluated data files. For the $^{10}\text{B}(n,\alpha)$ branching ratio, measurements were analysed and the results were published and presented at working party meetings. A new "set-up" was installed at GELINA and is presently being tested. These new results will be included into a new evaluation of the standards data file.

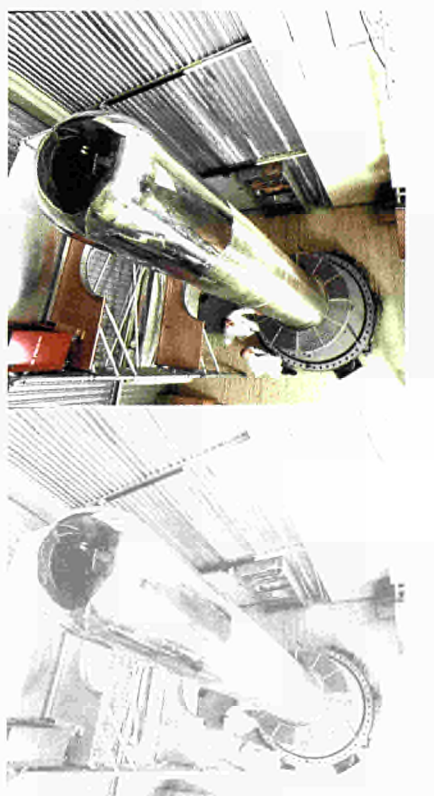
$^{10}\text{B}(n,\alpha\gamma)$ measurements await the completion of the characterisation of the ^{238}U samples. An ionisation chamber for this purpose was finalised.

Refurbishing of 7MV Van de Graaff Accelerator

In 1999, the 7MV Van de Graaff accelerator was refurbished, marking a 24 year gap since the accelerator was installed in 1976.

The belt was replaced for the first time in 24 years of operation which is exceptional considering that such the lifetime of such belts is normally 3-5 years. Furthermore the accelerator tube, the screen and the stabilization section were replaced.

As a result of this overhaul the VDG is now capable of delivering higher beam currents and a more stable high voltage.



The 7MV Van de Graaff accelerator - open for the first time in 24 years

Summer School

On May 17th - 21st, IRMM organised and hosted the Frédéric Joliot - Otto Hahn Summer School. 43 external visitors from 6 different countries attended. The Proceedings of the Summer School were published.

Primary Actinide Standard Data

Fission fragment yield data are of importance for the determination of delayed neutron production and decay heat calculation and hence, influence the reactivity of fast and thermal reactors. For fission fragment properties of $^{238}\text{U}(n, f)$, the analysis was finished, the numerical data were sent to the NEA database and the results were published.

For $^{239}\text{Pu}(n, f)$ in resonances, the data acquisition was finalized and the analysis of the data is ongoing with preliminary results been presented at several international conferences.

For neutron multiplicity and spectrum measurements, new analysis of the fission neutron spectrum of ^{252}Cf in terms of scission neutrons was discussed and published. The analysis within the collaboration with the International Science and Technology Center (ISTC), Russia on neutron multiplicity measurements of several primary standard isotopes, continued at Petersburg Nuclear Physics Institute, Gatchina, (PNPI), Russia. The final report is being prepared.

Minor Actinide Standard Data

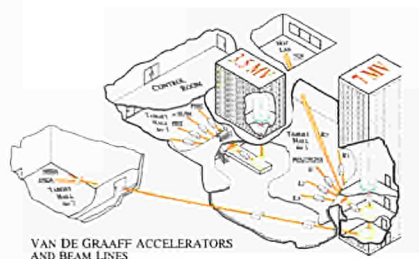
Minor actinides are important in the context of waste transmutation. Data re-analysis was completed for $^{237}\text{Np}(n, f)$, results were presented at international conferences and the final report is in preparation.

In collaboration with Institut Laue-Langevin (ILL), a ^{251}Cf -sample was received from Oak Ridge National Laboratory. Target preparation was conducted and completed in Mainz, Germany. The targets were sent to ILL, Grenoble and the experiment is scheduled for March 2000 in view of comparing results with the well-known $^{252}\text{Cf}(\text{SF})$ reaction.

Improvement and development of new methods also took place in 1999 with the commencement of a feasibility study for the use of the CAMAC-data acquisition system. A fast digitizer, capable of measuring the full signal information was tested and programmes for data acquisition were installed and improved.

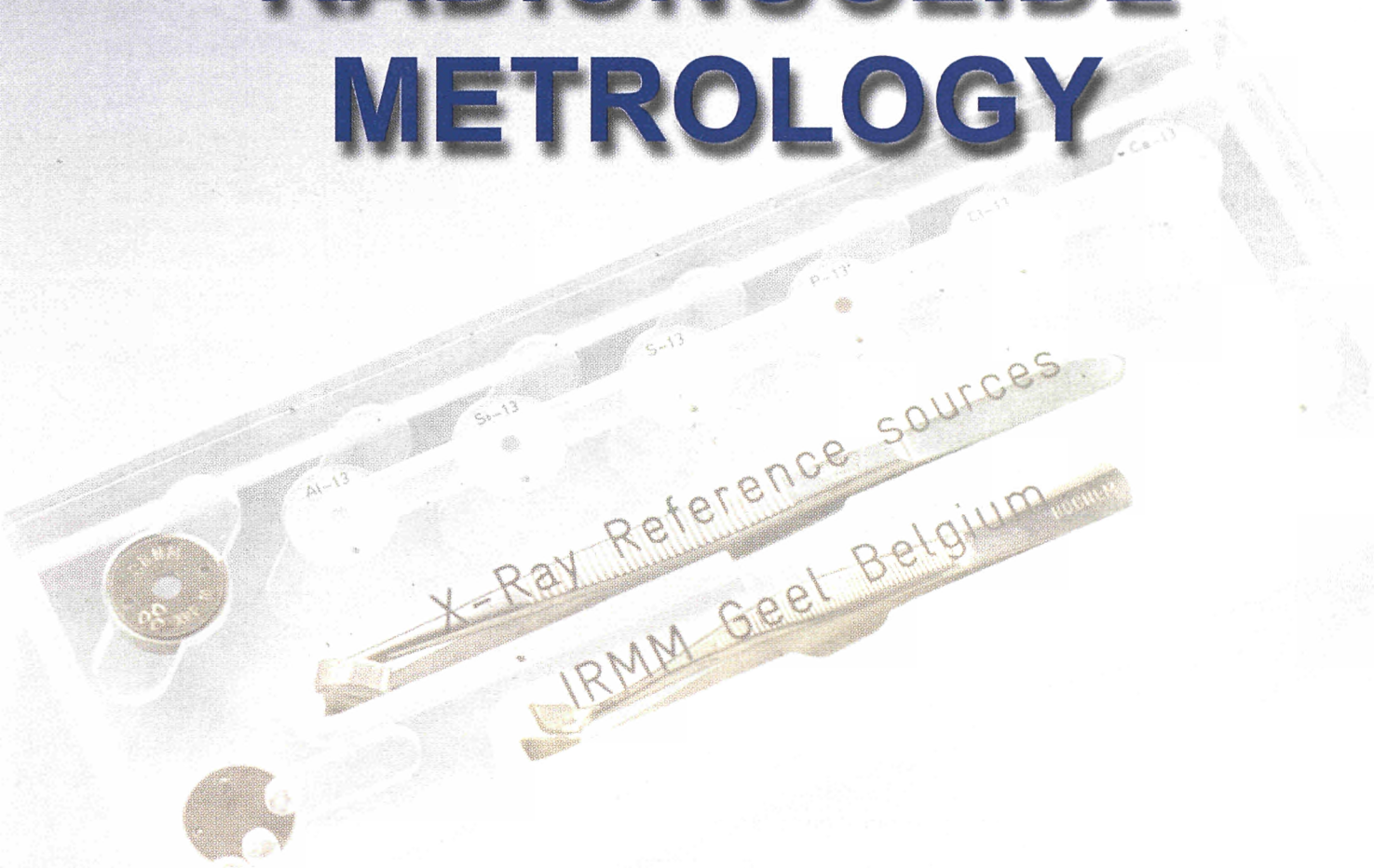
Gas Production in Nuclear Fuel

Neutron induced and spontaneous ternary fission is an important source of tritium production in nuclear reactors and nuclear fuel waste. If it comes into contact with living tissue the tritium can easily be absorbed. The dominantly produced ^4He , which is a metallurgical problem in fuel elements but is not radiotoxic, is used as a standard yield for tritium measurements. ^4He -yields have been determined for $^{239}\text{Pu}(n, f)$, $^{238,240,242,244}\text{Pu}(\text{SF})$ and $^{248}\text{Cm}(\text{SF})$; tritium measurements are in preparation.



Schematic of the Van de Graaff building showing the 3.5 and 7 MV accelerators with beam lines

REFERENCE LABORATORY FOR RADIONUCLIDE METROLOGY



REFERENCE LABORATORY FOR RADIONUCLIDE METROLOGY

Metrology has become a very important factor in international trade and hence for European competitiveness. As a consequence, there is a great need for Key Comparisons for the establishment of "International Equivalence". Additionally, reference materials need to be characterised and certified for their radioactive components. Furthermore, there is a need for accurate radioactivity measurements in nuclear medicine and radiotherapy applications where accuracy is directly correlated with the quality of the diagnosis and/or therapy. Another new field of Radionuclide Metrology applications is ultra-low level radioactivity measurements where the detection of smallest traces of radioactive matter needs to be assessed (e.g. for environmental safeguards, semiconductor industry, medicine, etc.).

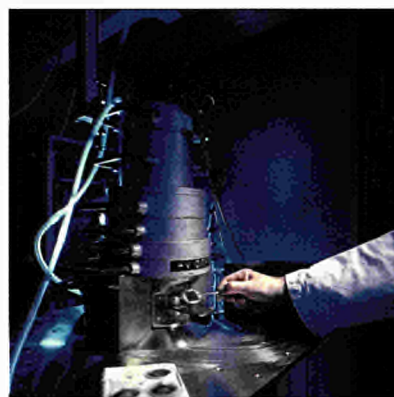
IRMM's Reference Laboratory for Radionuclide Metrology has been a leading laboratory for accurate radioactivity measurements for almost four decades now. It is a primary standards laboratory that realises the SI unit Becquerel for radionuclides in research, medicine, environment and industry and offers the following services and products:

- > Preparation of radioactive standard solutions and sources
- > High accuracy radioactivity measurements
- > Ultra low-level γ -ray spectrometry in a laboratory located 223 m underground
- > Radiochemical separations and characterisation of materials for their radioactive components and contents

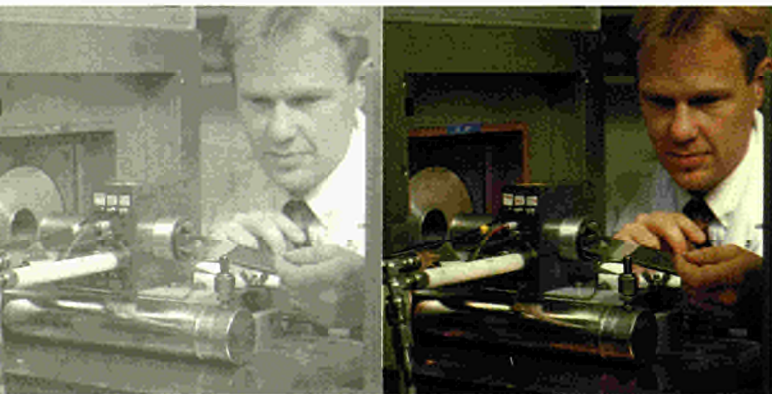
Furthermore, the RN Sector does accurate measurements of decay data, such as half lives, γ -ray and α -particle emission probabilities, fluorescence yields, and develops and improves radioactivity measurement methods.

Such work is carried out in close co-operation with other European primary standards laboratories under the auspices of EUROMET, the International Bureau of Weights and Measures (BIPM), the Consultative Ionising Radiation Committee (CCRI) of the Metre Convention, the International Committee for Radionuclide Metrology (ICRM) and the International Atomic Energy Agency (IAEA).

Additionally, the RN Sector co-operates with research laboratories and universities by offering its unique services in ultra low-level radioactivity measurement capabilities. The RN-Sector also provides special, highly accurate X-ray emitting sources for the calibration of detector systems, especially in the energy range between 1 keV and 6 keV.



Primary standardisation of radioactive sources by the $4\pi\beta$ - γ -coincidence method



Mikael Hult (SE) preparing a pressurised proportional counter for radioactivity measurements

FUTURE PROSPECTS

The Radionuclide Metrology sector will mainly concentrate on:

- > support to the international trading system through its primary standardisation work
- > pollution prevention and control through the characterisation and certification of reference materials for their radioactive components
- > medical and health applications through radioactivity measurements for diagnosis and radiotherapy

The participation in Key-Comparisons requested by BIPM or EUROMET in order to establish "International Equivalence" in measurement standards for the MRA (Mutual Recognition Arrangement) will undoubtedly grow. Additionally, requests for reference materials (food or fodder, soil, sediments and others) characterised for their radioactive components will certainly increase in the near future. It is also setting-up a QA system on the basis of the European Foundation for Quality Management (EFQM) to comply with Appendix B of the MRA.



CONTACT

Dietmar F.G. Reher, MSc.
Tel. +32 (0)14 571 263
Fax. +32 (0)14 584 273
dietmar.reher@irmm.jrc.be

MAJOR SCIENTIFIC RESULTS IN 1999

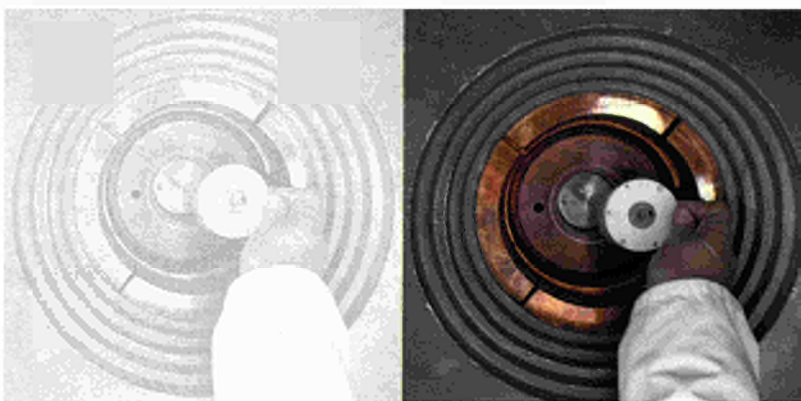
NIST Ocean Shellfish Reference Material

An ocean shellfish material will be issued by the National Institute for Standards and Technology (NIST), USA, as an environmental reference material. In collaboration with NIST, IRMM is participating in the characterisation of the ocean shellfish material for its radioactive components. The gamma-ray spectrometric measurements in the underground laboratory HADES and the analysis of the data have been completed. A chemical procedure has been developed for the separation of Sr and the actinides and will be used for the isolation of those elements from the ocean shellfish material and their subsequent assessment by liquid scintillation and alpha particle counting.

Swipe Samples for DG Energy and Transport

The HADES underground laboratory is ideally suited for measurements of swipe samples from nuclear installations with very low activity. The samples are collected by the EURATOM Inspectorate in Luxembourg and give vital information on nuclear activities. The aim is to enable the detection of illegal nuclear activities.

Two batches of samples were measured in HADES and analysed in 1999. The EGS4 Monte-Carlo code was used for simulating hot spots and radioactivity concentration variations within the samples as well as simulations for coincidence summing corrections.



In the underground laboratory, José Das Neves (PT) places a swipe sample into the ultra low level Ge spectrometer to assess traces of unauthorised nuclear material

Food and Environmental Samples

A project on the dating of soil and river sediment took place in 1999. Samples were collected downstream from a German U-mine to yield information on sedimentation rates and the burden on the environment. Five samples were measured in HADES and the data were evaluated and published.

Additionally, a project has started on milk powder and soil RMs. Test samples were provided by the International Atomic Energy Agency (IAEA) and extractions of Sr, Am, Pu, U and Th using extraction resins have been tried out.

BIPM/CCRI - the Bq at the Basic Level - Part-1

In October 1999, the MRA of the Metre Convention was signed at the BIPM. The signatories of this agreement, to which IRMM belongs, mutually accept NMI's calibration and measurement certificates. This is an important historical step for international trade. Central in this MRA are the so-called "key-comparisons", carried out under the auspices of the BIPM, its Consultative Committees (CCs) or Regional Metrology Organisations, such as EUROMET. In radioactivity the most important "Key-comparison" is SIR, the *Système International de Référence*. It consists of a commercial ionisation chamber and has been calibrated with about 500 world-wide primary standards of radioactivity for about 60 radionuclides. The estimated effort world-wide for this is about 200 man-years, which corresponds to about 10 - 20 million EUR.

BIPM/CCRI - the Bq at the Basic Level - Part-2

IRMM, in collaboration with NPL, launched a project for CCRI to rescue the SIR in case it would fail to operate. An ionisation chamber that is reproducible in its characteristics has been conceived at IRMM. Simulations for parameters, such as physical dimensions, materials, type and pressure of gas, were carried out by means of EGS4. Comparison with the efficiency of existing chambers was successful. A first draft design is being prepared and a patent has been filed.

Zn Impurities in GaAs

It is of utmost importance to control the levels of impurities in semiconductor samples. Because of the minute quantities of many impurities the measure-

ments required to achieve adequate control may be difficult to realise. IRMM co-operated with the SME "Freiberger Compound Materials" Germany in order to develop a highly sensitive technique for determining Zn impurities in GaAs-wafers. The technique which was developed proved highly useful and comprised of three major steps. First, neutron activation in a reactor, secondly chemical separation at IRMM and finally ultra low-level gamma-ray spectrometry in HADES. The technique can detect Zn concentrations of about 20 pg/g in GaAs which is 10 times better than requested.

Environmental Neutron Fluence

At IRMM a new way of measuring very low neutron fluxes is being investigated. Discs of metals with high neutron cross sections are placed at the location of the study. The discs are later measured using ultra low-level gamma-ray spectrometry in HADES. Several important applications evolved during 1999:

- (A) Important data on cosmogenic production rates in different materials is being systematically collected.
- (B) The neutron flux in the BR1 reactor hall of the Studiecentrum voor Kernenergie was mapped in order to fully implement the ALARA principle.
- (C) Discs were placed around the IRMM buildings containing artificial neutron sources for radioprotection purposes and
- (D) Retrospective assessment of the neutron flux in buildings near the JCO-site in Japan where there was an uncontrolled nuclear chain reaction on September 30th, 1999, has begun.



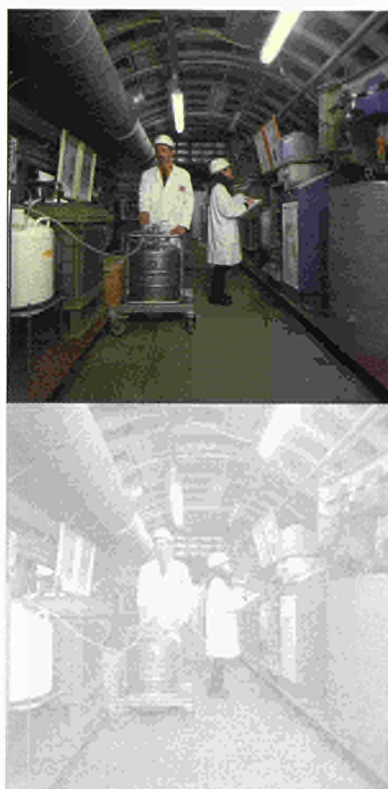
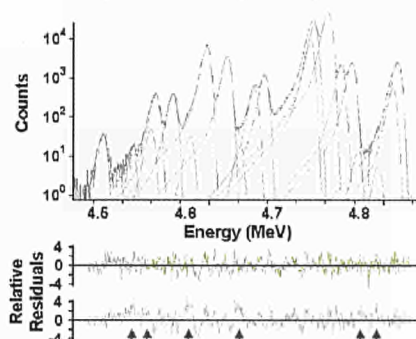
Lena Johansson (SE) drying a radioactive solution using the new source drying system

^{237}Np Measurements

The study of ^{237}Np is very important because it is produced in nuclear power plants, has a long half-life (~ 2 million years), and will be one of the most important radionuclides in final repositories.

In the laboratory, a solution of ^{237}Np has been standardised (in the frame of EUROMET project 416) by the defined solid angle α -counting method and additionally measurements of α -particle and γ -ray spectra were performed. The α -particle measurements led to the discovery of six new α -particle transitions in the decay scheme of ^{237}Np . The evaluation of the γ -ray spectra is ongoing.

α -particle spectrum of ^{237}Np



José Das Neves (PT) and María Martínez (ES) in the HADES ultra low level laboratory

Multiple Tasks

IRMM is part of the BOREXINO collaboration, which aims at building a gigantic detector for neutrino detection in the world's largest underground laboratory in Gran Sasso, Italy. The demands for radiopurity are extreme and were thought, by many, to be impossible to realise some years ago. However, with scientific developments, it now seems that the detector will be in operation in 2001. Many materials need to be investigated for their radiopurity before put into the detector and in this context IRMM has contributed by carrying out ultra low-level measurements of materials in HADES.

In the frame of the "MRA" the evaluation of the key-comparison SIR was continued; a first set of AeRef values was calculated, distributed to members of the working group and made ready for the International Equivalence calculations.

Finally for the EUROMET project 428 which addresses the transfer of Ge detector efficiency calibration, the exercise has been completed and the results of the IRMM Monte Carlo calculations were in good agreement with the experimental values.









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